Dinitrogen chemistry from trigonally coordinated iron and cobalt platforms

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Experimental Section

All manipulations were carried out using standard Schlenk or glove-box techniques under a dinitrogen atmosphere. Unless otherwise noted, solvents were deoxygenated and dried by thorough sparging with N_2 gas followed by passage through an activated alumina column. Non-halogenated solvents were typically tested with a standard purple solution of sodium benzophenone ketyl in tetrahydrofuran in order to confirm effective oxygen and moisture removal. Deuterated solvents were degassed and stored over activated 3-Å molecular sieves prior to use. THF-d_8 was dried by passage over activated alumina and stored over activated sieves prior to use. $[PhBP^{iPr}_3][Tl]$, $[PhBP^{iPr}_3]FeCl$, and $[PhBP^{iPr}_3]CoI$ were prepared as previously reported.[1] $[Fc][BPh_4]$[2] and *p*-tolylazide[3] were prepared according to literature procedures. Adamantylazide (Aldrich), chlorotrimethylsilane (Strem), and methyl tosylate (Aldrich) were used as received without further purification. ^{15}N-Labeled N_2 gas was purchased from Cambridge Isotope Laboratory. All reagents were purchased from commercial vendors and used without further purification unless explicitly stated. Elemental analyses were carried out at Desert Analytics, Tucson, Arizona. NMR spectra were recorded at ambient temperature on Varian Mercury 300 MHz, Joel 400 MHz, and an Inova 500 MHz spectrometers, unless otherwise noted. ^1H NMR chemical shifts were referenced to residual solvent. ^{31}P NMR chemical shifts are reported relative to an external standard of 85% H_3PO_4. IR spectra were recorded on a Bio-Rad Excalibur FTS 3000 spectrometer controlled by Win-IR Pro software. UV-vis measurements were taken on a Hewlett Packard 8452A diode

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array spectrometer using a quartz crystal cell with a Teflon cap. X-ray diffraction studies were carried out in the Beckman Institute Crystallographic Facility on a Bruker Smart 1000 CCD diffractometer.

X-ray Crystallography Procedures. X-ray quality crystals were grown as indicated in the experimental procedures for each complex. The crystals were mounted on a glass fiber with Paratone-N oil. Structures were determined using direct methods with standard Fourier techniques using the Bruker AXS software package. In some cases, Patterson maps were used in place of the direct methods procedure.

{[PhBP$^{iPr}_3$]Fe(N_2)}{MgCl($THF)_2$} (**3a**): A solution of [PhBP$^{iPr}_3$]FeCl (**1**) (100 mg, 0.175 mmol) in THF (2 mL) was added to a stirring suspension of Mg^0 powder (100 mg, 4.1 mmol) in THF (2 mL) at room temperature. After stirring for 12 h, the resulting orange-amber solution was filtered, then evaporated to dryness in vacuo. The brown solids were dissolved in THF (2 mL) and the solution was filtered once again. Vapor diffusion of petroleum ether into the THF filtrate resulted in the precipitation of brown solids. This brown solid was dried under vacuum to afford analytically pure product (113 mg, 84%). ^{1}H NMR (THF-d_8, 300 MHz): δ 26.8, 18.07 (bs), 13.5, 12.4, 3.53 (THF), 1.73 (THF), -6.35 (bs). UV-vis (THF) λ_{max}, nm (ε, M^{-1} cm^{-1}): 373 (3200), 909 (620). Evans Method (THF-d_8): 2.92 μ_B. IR: (THF/KBr) ν_{NN} = 1830 cm^{-1}. Anal. Calcd for $C_{35}H_{69}BClFeMgN_2O_2P_3$: C, 54.65; H, 9.04; N, 3.64. Found: C, 54.60; H, 9.19, N, 3.33.

{[PhBP$^{iPr}_3$]Fe($^{15}N_2$)}{MgCl(THF)$_2$} (**3a-$^{15}N_2$**) A solution of **1** (220 mg, 0.38 mmol) in THF (10 mL) was added to a suspension of Mg powder (100 mg, 4.1 mmol) in THF (20 mL) under a counter-flow of Argon gas into a 200 mL sealable Schlenk bomb reactor in the dry box. The headspace of the bomb was then evacuated under vacuum. The solution was degassed via three freeze-pump-thaw cycles and then exposed to one atmosphere of labeled $^{15}N_2$ gas (ca. 8.3 mmol). The reaction was stirred for 24 h at room temperature, after which time the solution turned a dark red brown. The reaction work-up followed the same protocol as that described for **3a** (233 mg, 79%). Spectroscopic features in the ^{1}H NMR were identical to those reported for **3a**. IR: (THF/KBr) ν_{NN} = 1769 cm^{-1}.

{[PhBP$^{iPr}_3$]Fe(N$_2$)}$_2$Mg(18-C-6) (**3b**): A solution of (**1**) (45 mg, 0.078 mmol) in THF (2 mL) was added to a stirring suspension of Mg^0 powder (100 mg, 4.1 mmol) in THF (2 mL) at room temperature. After stirring for 16 h, the resulting orange-amber solution was filtered to remove excess Mg^0 and insoluble residue. A solution of 18-crown-6 (24 mg, 0.091 mmol) in THF (1 mL) was added to the orange-amber filtrate and the solution was stirred for 2 h at room temperature. This solution was then filtered to remove a white precipitate. The volatiles were evaporated in vacuo to yield a brown solid that was then washed with petroleum ether (2 x 3 mL). The brown solid was dried under vacuum to afford analytically pure product (92 mg, 82.5%). IR: (THF/KBr) ν_{NN} = 1884 cm^{-1}. Anal. Calcd for $C_{66}H_{130}B_2Fe_2MgN_4O_6P_6$: C, 55.86; H, 9.23; N, 3.95. Found: C, 55.69; H, 9.32; N, 3.99.

{[PhBP$^{iPr}_3$]Fe($^{15}N_2$)}$_2$Mg(18-C-6) (**3b-$^{15}N_2$**): A solution of **3a-$^{15}N_2$** (20 mg, 0.026 mmol) in THF (2 mL) was added to a solution of 18-crown-6 (13.7 mg, 0.05 mmol) in THF (1 mL) under a blanket of Argon gas in a 25 mL Schlenk round-bottom flask at room temperature. The reaction was stirred for 30 min at room temperature. The reaction work-up followed the same protocol as that described for **3b**. Spectroscopic features in the ^{1}H NMR were identical to those reported for **3b**. IR: (THF/KBr) ν_{NN} = 1822 cm^{-1}.

{[PhBP$^{iPr}_3$]Co(N$_2$)}$_2${Mg(THF)$_4$} (**4a**): A solution of [PhBP$^{iPr}_3$]CoI (**2**) (100 mg, 0.15 mmol) in THF (2 mL) was added to a stirring suspension of Mg^0 powder (100 mg, 4.1 mmol) in THF (2 mL) at room temperature. After stirring for 12 h, the resulting red solution was filtered and then evaporated to dryness in vacuo. The red solids were dissolved in THF (2 mL) and the solution was filtered once again. Vapor diffusion of petroleum ether into the THF filtrate resulted in the precipitation of large red crystals. Suitable crystals were selected for an X-ray diffraction study. The red crystals were isolated by decanting the remaining solution and drying under vacuum to afford analytically pure product (169 mg, 78%). ^{1}H NMR (THF-d_8, 300 MHz): δ 18.5 (bs), 13.6, 12.1, 11.8, 6.3, 3.58 (THF), 1.73 (THF), 0.9, -19.5 (bs). UV-vis (THF) λ_{max}, nm (ε, M^{-1} cm^{-1}): 985 (580). Evans Method (THF-d_8): 2.01 μ_B. IR: (THF/KBr) ν_{NN} = 1863 cm^{-1}. Anal. Calcd for $C_{70}H_{138}B_2Co_2MgN_4O_4P_6$: C, 58.00; H, 9.60, N, 3.87. Found: C, 57.41; H, 9.75; N 3.50.

{[PhBP$^{iPr}_3$]Co($^{15}N_2$)}$_2${Mg(THF)$_4$} (**4a-$^{15}N_2$**): A solution of **2** (200 mg, 0.30 mmol) in THF (10 mL) was added to a suspension of Mg powder (100 mg, 4.1 mmol) in THF (20 mL) under a counter-flow of Argon gas into a 100 mL sealable Schlenk bomb reactor in the dry box. The headspace of the bomb was then evacuated under vacuum. The solution was degassed via three freeze-pump-thaw cycles and then exposed to one atmosphere of labeled $^{15}N_2$ gas (ca. 4.1 mmol). The reaction was stirred for 24 h at room temperature, after which time the solution turned a dark red color. The reaction work-up followed the same protocol as that described for **4a** (181 mg, 87%). The product was analyzed and proved identical to **4a** according to its ^{1}H NMR. IR: (THF/KBr) ν_{NN} = 1802 cm^{-1}.

{[PhBP$^{iPr}_3$]Co(N_2)}$_2$Mg(18-C-6) (**4b**): A solution of [PhBP$^{iPr}_3$]CoI (**2**) (52 mg, 0.078 mmol) in THF (2 mL) was added to a stirring suspension of Mg^0 powder (100 mg, 4.1 mmol) in THF (2 mL) at room temperature. After stirring for 10 h, the resulting red-orange solution was filtered. A solution of 18-crown-6 (21 mg, 0.078 mmol) in THF (1 mL) was added to the red-orange filtrate. The resulting solution was stirred for 45 minutes, during which time the color changed to an intense red-orange. The volatiles were removed in vacuo to afford a red-orange solid that was then washed with petroleum ether (2 x 3 mL). The resulting residue was dried under vacuum to afford analytically pure material (101 mg, 91%). IR: (THF/KBr) ν_{NN} = 1896 cm^{-1}. Anal. Calcd for $C_{66}H_{130}B_2Co_2MgN_4O_6P_6$: C, 55.61; H, 9.19; N, 3.93. Found: C, 55.60; H, 9.42; N, 3.88.

{[PhBP$^{iPr}_3$]Co($^{15}N_2$)}$_2$Mg(18-C-6) (**4b-$^{15}N_2$**): A solution of **4a-$^{15}N_2$** (20 mg, 0.014 mmol) in THF (2 mL) was added to a solution of 18-crown-6 (7.3 mg, 0.03 mmol) in THF (1 mL) under a blanket of Argon gas in a 25 mL Schlenk round-bottom flask at room temperature. The reaction was stirred for 30 min at room temperature. The reaction work-up followed the same protocol as that described for **4b**. The product was analyzed and proved identical to **4b** according to its ^{1}H NMR. IR: (THF/KBr) ν_{NN} = 1842 cm^{-1}.

{[PhBP$^{iPr}_3$]Fe}$_2$(μ-N$_2$) (**5**): A 0.5 weight % Na/Hg amalgam (2.8 mg, 0.132 mmol of sodium dissolved in 560 mg of mercury) was stirred in THF (5 mL) for several minutes. A solution of **1** (75 mg, 0.131 mmol) in THF (1 mL) was added to the vigorously stirring amalgam at room temperature under nitrogen. After stirring for 4 h, the resulting brown solution was filtered (filter paper/pipet) to remove insoluble residues. The volatiles were then removed in vacuo. The resulting brown solids were dissolved in THF (3 mL), to which petroleum ether was then added (2 mL). Storing this solution at -33 ºC for 20 h resulted in the precipitation of an amorphous brown solid. These solids were isolated by decanting the remaining solution, and then washed with petroleum ether (2 x 3 mL). The isolated solids were dried under a blanket of nitrogen (in the glove box) to afford analytically pure material (127 mg, 88%). Note: the isolated solids are highly unstable to benzene, which caused decomposition of this material to as yet unidentified products. ^{1}H NMR (THF-d_8, 300 MHz): δ 26.83 (s), 18.13 (bs), 13.50, 12.36, 7.30, 0.85, -5.02, -6.30 (bs). UV-vis (THF) λ_{max}, nm (ε, M^{-1} cm^{-1}): 372 (6700), 905 (1200). Evans Method

(THF-d_8): 4.12 μ_B (calculated per Fe center). Anal. Calcd for $C_{54}H_{106}B_2Fe_2N_2P_6$: C, 58.82; H, 9.69; N, 2.54. Found: C, 58.88; H, 9.88; N, 2.38.

Synthesis of 5 by oxidation of 3a: A solution of **3a** (25 mg, 0.039 mmol) in THF (3 mL) was added in two portions to a stirring slurry of $[Cp_2Fe][BPh_4]$ (19.7 mg, 0.039 mmol) in THF (3 mL) at room temperature. The color of the solution progressed from the red-amber of **3a** to a darker brown color over the period of 6 h. This color change was accompanied by the disappearance of the ν_{NN} band (1830 cm^{-1}) of **3a**. After this time, the precipitate was filtered to afford a brown filtrate. Upon isolation by removal of solvent in vacuo, the product was analyzed and proved identical to **5** according to its ^1H NMR, UV-Vis spectrum, and independent combustion analysis. Several diamagnetic products (less than 10% by ^1H NMR) also formed during this reaction; thus the reduction of **1** by sodium-amalgam provides a cleaner synthesis of **5**.

$\{[PhBP^{iPr}_3]Co\}_2(\mu\text{-}N_2)$ (6): A 0.5 weight % Na/Hg amalgam (2.0 mg, 0.087 mmol of sodium dissolved in 420 mg of mercury) was stirred in THF (5 mL). A solution of **2** (60 mg, 0.090 mmol) in THF (1 mL) was added to the amalgam at room temperature. After stirring for 6 h, the resulting pale rose solution was filtered to remove insoluble residues. Petroleum ether (2 mL) was added drop-wise to the THF solution. The solution was then stored at -33 ºC for 10 h which resulted in the deposition of large pale purple crystals. The crystals were isolated by decanting the remaining solution and allowing the crystals to dry under a blanket of nitrogen (in the glove box), affording analytically pure material (92 mg, 92%). Note: the isolated crystals proved unstable to benzene, which caused

decomposition to as yet unidentified products. ^{1}H NMR (THF-d_8, 300 MHz): δ 17.34 (bs), 6.63, 6.36, 0.91 (bs). UV-vis (THF) λ_{max}, nm (ε, M^{-1} cm^{-1}): 745 (170), 948 (270). Evans Method (THF-d_8): 3.01 μ_B (calculated per Co center). Anal. Calcd for $C_{54}H_{106}B_2Co_2N_2P_6$: C, 58.50; H, 9.64; N, 2.53. Found: C, 57.88; H, 9.72; N, 2.45.

Synthesis of 6 by oxidation of 4a: A solution of **4a** (30 mg, 0.021 mmol) in THF (3 mL) was added in two portions to a stirring slurry of [Fc][BPh_4] (10.5 mg, 0.021 mmol) in THF (3 mL) at room temperature. The color of the solution progressed from the cherry-red of **4a** to a turbid, colorless solution over the period of 1 h. The color change was accompanied by the disappearance of the ν_{NN} (1860 cm^{-1}) of **4a** by IR. After this time, the precipitate was filtered to provide a pale rose filtrate. The product of the oxidation was identical to **6** as ascertained by ^{1}H NMR and UV-vis spectroscopies, and an independent combustion analysis.

[{[PhBP$^{iPr}_3$]Fe}$_2$(μ-N$_2$)][Na(THF)$_6$] (**7**): A 0.5 weight % Na/Hg amalgam (15 mg, 0.65 mmol of sodium dissolved in 3 g of mercury) was stirred in THF (5 mL) for several minutes. A solution of **1** (75 mg, 0.131 mmol) in THF (1 mL) was added to the vigorously stirring amalgam at room temperature. After stirring for 13 h, the resulting red-brown solution was filtered (filter paper/pipet) to remove insoluble residues. The THF solution was then slowly layered with petroleum ether (2.5 mL) so as to just begin precipitation of the salts. The slightly turbid mixture was then re-dissolved by shaking and allowed to stand for 48 h at room temperature. Dark brown crystals deposited during this time that were isolated by decanting the remaining solution. A suitable crystal was

selected for an X-ray diffraction study. The crystals were dried in vacuo to afford analytically pure material (189 mg, 93%). ^{1}H NMR (THF-d_8, 300 MHz): δ 25.8, 16.10 (bs), 13.22, 12.10, 9.47 (bs), 3.64 (THF), 1.85 (THF), -6.84 (bs). UV-vis (THF) λ_{max}, nm (ε, M^{-1} cm^{-1}): 374 (2800), 905 (570). Anal. Calcd for $C_{78}H_{154}B_2Fe_2N_2NaO_6P_6$: C, 60.12; H, 9.96; N, 1.80. Found: C, 59.61; H, 10.22; N 1.82.

[{[PhBP$^{iPr}_3$]Co}$_2$(μ-N$_2$)][Na(THF)$_6$] (**8**): A 0.5 weight % Na/Hg amalgam (9 mg, 0.39 mmol of sodium dissolved in 1.8 g of mercury) was stirred in THF (5 mL) for several minutes. A solution of **2** (50 mg, 0.075 mmol) in THF (1 mL) was then added to the vigorously stirring amalgam at room temperature. After stirring for 12.5 h, the resulting red-brown solution was filtered to remove insoluble material. The THF solution was then layered with petroleum ether (2.5 mL), which slowly caused some solids to precipitate. The turbid mixture was then re-dissolved by thorough shaking and allowed to stand for 24 h at room temperature. Dark red-brown crystals deposited during this time that were then isolated by decanting the supernatant. A suitable crystal was selected for an X-ray diffraction study. The crystals were dried in vacuo to afford analytically pure material (104 mg, 89%). ^{1}H NMR (THF-d_8, 300 MHz): δ 24.23, 18.48 (bs), 16.85 (bs), 12.12, 11.56, 3.58 (THF), 1.73 (THF), 0.14, -1.97 (bs), -3.62, -4.53, -19.45 (bs). UV-vis (C_6H_6) λ_{max}, nm (ε, M^{-1} cm^{-1}): 566 (210), 969 (450). Anal. Calcd for $C_{78}H_{154}B_2Co_2N_2NaO_6P_6$: C, 59.89; H, 9.92; N, 1.79. Found: C, 59.57; H, 9.64; N, 1.84.

[PhBP$^{iPr}_3$]Fe≡NAd (**9**): Complex **5** was generated in situ by the following procedure: A 0.5 weight % Na/Hg amalgam (3.6 mg, 0.157 mmol of sodium dissolved in 720 mg of

mercury) was stirred in THF (5 mL). A solution of **1** (60 mg, 0.105 mmol) in THF (1 mL) was added to the amalgam at room temperature. After stirring for 8 h, the resulting red-brown solution was filtered to remove insoluble material, providing a solution of **5**. A solution of adamantylazide (20.2 mg, 0.105 mmol) in THF (0.5 mL) was then added to this red-brown solution. The resulting solution was stirred at room temperature for 28 h, during which time the solution color turned to a deep red. The solution was filtered, layered with petroleum ether (2 mL), and stored at -33 ºC for 36 h. Opaque red crystals deposited during this time and were isolated by decanting the remaining liquor. A suitable crystal was selected for an X-ray diffraction study. The crystals were dried in vacuo to afford analytically pure material (49.6 mg, 69%). ^{1}H NMR (C_6D_6, 300 MHz): δ 56.4, 38.4, 20.2, 13.1, 12.2, 9.45, 9.15, 5.65 (dd), 2.76, -0.78, -5.93, -7.13, -31.4. Evans Method (C_6D_6): 1.94 BM. Anal. Calcd for $C_{37}H_{68}BFeNP_3$: C, 64.73; H, 9.98; N, 2.04. Found: C, 65.13; H, 10.08; N, 1.98.

[PhBP$^{iPr}_3$]Co≡N-*p*-tol (**10**): Complex **6** was generated in situ by the following procedure: A 0.5 weight % Na/Hg amalgam (1.4 mg, 0.06 mmol of sodium dissolved in 276 mg of mercury) was stirred in THF (3 mL). A solution of **2** (40 mg, 0.060 mmol) in THF (1 mL) was added to the amalgam at room temperature. After stirring for 6 h, the resulting pale-rose solution was filtered (filter-paper/pipet) to remove insoluble material. A solution of *p*-tolylazide (8 mg, 0.06 mmol) in THF (0.5 mL) was then added to the pale-rose colored solution, which caused a dramatic color change to a deep red. The solution was stirred for 1 h and the volatiles were then removed in vacuo. The resulting red solid was washed with petroleum ether (3 x 5 mL). Crystals of **10** were grown by

extracting the red solid into benzene and slowly allowing petroleum ether to diffuse into this solution. The red crystals that formed were isolated by decanting off the remaining solution and drying in vacuo (36 mg, 93%). Suitable crystals were selected for an X-ray diffraction study. ^{1}H NMR (C_6D_6, 300 MHz): δ 7.90 (d, J = 7.8 Hz, 2H), 7.73 (d, J = 7.5 Hz, 2H), 7.54 (t, J = 6.9 Hz, 2H), 7.31 (t, J = 7.8 Hz, 1H), 6.63 (d, J = 7.5 Hz, 2H), 2.00 (bs, 6H), 1.58 (s, 3H), 1.43 (dd, J = 5.4, 12.6 Hz, 18H), 1.21 (bs, 18H), 0.50 (bs, 6H). ^{31}P {^{1}H} NMR (C_6D_6, 121.4 MHz): δ 85 (bs). Anal. Calcd for $C_{34}H_{60}BCoNP_3$: C, 63.26; H, 9.37; N, 2.17. Found: C, 63.18; H, 9.36; N, 2.08.

[PhBP$^{iPr}_3$]Fe(N$_2$Me) (**11**): A solution of **1** (75 mg, 0.131 mmol) in THF (2 mL) was added to a stirring suspension of Mg^0 powder (100 mg, 4.1 mmol) in THF (2 mL) at room temperature to generate **3a** in situ. After stirring for 12 h, the resulting orange-amber solution was filtered (filter-paper/pipet) to remove excess Mg powder and insolubles. A solution of methyl tosylate (24.4 mg, 0.131 mmol) in THF (2 mL) was added in two portions to the stirring solution of "[FeN_2--Mg]" at room temperature. The combined solution was stirred for 6 h, then the solution was filtered (filter-paper/pipet) and evaporated to dryness in vacuo. The resulting oily brown solid was extracted into benzene and filtered (filter-paper/pipet) to yield a gold solution. The benzene was removed in vacuo to afford a tan brown solid. The solids were washed with petroleum ether (2 x 3 mL) and dried in vacuo to afford analytically pure material (38 mg, 56%). ^{1}H NMR (C_6D_6, 300 MHz): δ 34, 19.2, 18.5, 3.2 (bs), 2.0 (bs), -14 (bs), -30 (bs). Evans Method (C_6D_6): 4.93 μ_B. IR: (THF/KBr) ν_{NN} = 1597 cm^{-1}. Anal. Calcd for

$C_{28}H_{56}BFeN_2P_3$: C, 57.95; H, 9.73; N, 4.83. Found: C, 58.65; H, 9.98; N, 4.78. *Note*: An isolated sample of **3a** also reacted with stoichiometric MeOTs in THF to form **11**.

[PhBP$^{iPr}_3$]Fe($^{15}N_2$Me) (11-$^{15}N_2$): A solution of methyl tosylate (24.1 mg, 0.131 mmol) in THF (2 mL) was added in two portions to the stirring solution of **3a-$^{15}N_2$** (50 mg, 0.065 mmol) in THF (2 mL) under a blanket of Argon in a 25 mL round-bottom Schlenk flask. The reaction was stirred for 2 h at room temperature and worked-up in a similar fashion to **11**. The product was analyzed and proved identical to **11** according to its ^{1}H NMR spectrum. IR: (THF/KBr) ν_{NN} = 1538 cm^{-1}.

[PhBP$^{iPr}_3$]Co(N_2Me) (12): A solution of **2** (100 mg, 0.15 mmol) in THF (2 mL) was added to a stirring suspension of Mg^0 powder (100 mg, 4.1 mmol) in THF (2 mL) at room temperature to generate **4a** in situ. After stirring for 12 h, the resulting red solution was filtered (filter-paper/pipet). A solution of methyl tosylate (27.9 mg, 0.15 mmol) in THF (2 mL) was added dropwise to the stirring solution of "[$(CoN_2)_2$--Mg]" at room temperature. The solution was stirred for 5 h where the color changed from cherry-red to brown, then the solution was filtered (filter-paper/pipet) and evaporated to dryness under vacuum. The resulting brown solid was extracted into benzene and filtered (filter-paper/pipet) to yield a tan solution. The benzene was removed in vacuo to afford a tan solid. The solids were washed with petroleum ether (2 x 3 mL) and dried in vacuo to afford analytically pure material (60 mg, 68%). ^{1}H NMR (C_6D_6, 300 MHz): δ 23.9 (bs), 12.8, 3.22 (bs), 1.04 (bs). Evans Method (C_6D_6): 1.92 μ_B. IR: (THF/KBr) ν_{NN} = 1599 cm^{-1}. Anal. Calcd for $C_{28}H_{56}BCoN_2P_3$: C, 57.64; H, 9.67; N, 4.80. Found: C, 57.75; H,

9.72; N, 4.69. *Note*: An isolated sample of **4a** also reacted with stoichiometric MeOTs in THF to form **12**.

[PhBP$^{iPr}_3$]Co($^{15}N_2$Me) (**12-$^{15}N_2$**): A solution of methyl tosylate (7.7 mg, 0.041 mmol) in THF (2 mL) was added in two portions to a stirring solution of **4a-$^{15}N_2$** (50 mg, 0.034 mmol) in THF (2 mL) under a blanket of Argon in a 25 mL round-bottom Schlenk flask. The reaction was stirred for 4 h at room temperature and worked-up in a similar fashion to **12**. The spectroscopic features in the ^{1}H NMR spectrum were identical to those reported for **12**. IR: (THF/KBr) ν_{NN} = 1542 cm^{-1}.

[PhBP$^{iPr}_3$]Co(N_2SiMe$_3$) (**13**): A solution of **2** (100 mg, 0.15 mmol) in THF (2 mL) was added to a stirring suspension of Mg^0 powder (100 mg, 4.1 mmol) in THF (2 mL) at room temperature to generate **4a** in situ. After stirring for 12 h, the resulting red solution was filtered (filter-paper/pipet). A solution of chlorotrimethylsilane (16.3 mg, 0.15 mmol) in THF (2 mL) was added dropwise to the stirring solution at room temperature. The resulting solution was stirred for 5 h, during which time the color changed from cherry-red to brown. The solution was then filtered and evaporated to dryness under vacuum. The resulting brown solid was extracted into benzene and filtered to provide a brown filtrate. The volatiles were removed in vacuo to afford brown solids that were washed with petroleum ether (2 x 3 mL) and then dried in vacuo to afford analytically pure material (76 mg, 79%). ^{1}H NMR (C_6D_6, 300 MHz): δ 24.1 (bs), 13.1, 3.22 (bs), 0.15 (bs). Evans Method (C_6D_6): 2.08 μ_B. IR: (THF/KBr) ν_{NN} = 1654 cm^{-1}. Anal. Calcd for $C_{30}H_{62}BCoN_2P_3Si$: C, 56.16; H, 9.74; N, 4.37. Found: C, 56.45; H, 9.83; N, 4.22.

Alternative synthesis of 13: A 0.5 weight % Na/Hg amalgam (8.3 mg, 0.36 mmol of sodium dissolved in 1.66 mg of mercury) was stirred in THF (3 mL). Chlorotrimethylsilane (39 mg, 0.36 mmol) was added directly to the stirring solution of amalgam prior to the addition of **2**. A solution of **2** (40 mg, 0.060 mmol) in THF (1 mL) was added to the amalgam/Me_3SiCl solution at room temperature. The combined solutions were stirred for 5 h, after which time the solution was filtered to remove mercury and precipitates. The silyldiazenido product **13** was identified by IR and ^1H NMR spectroscopies, and the dinuclear, N_2-bridged product **6** was also identified. The ratio of **13** to **6** was 85:15 according to integration of a ^1H NMR spectrum of the crude product mixture.

[PhBP$^{iPr}_3$]Co($^{15}N_2SiMe_3$) (**13-$^{15}N_2$**): A solution of chlorotrimethylsilane (3.6 mg, 0.034 mmol) in THF (2 mL) was added in two portions to a stirring solution of **4a-$^{15}N_2$** (25 mg, 0.017 mmol) in THF (2 mL) under a blanket of Argon in a 25 mL round-bottom Schlenk flask. The reaction was stirred for 4 h at room temperature and worked- up in a similar fashion to **13**. The product was analyzed and proved identical to **13** according to its ^1H NMR spectrum. IR: (THF/KBr) ν_{NN} = 1596 cm^{-1}.

Figure 1. Fully labeled drawing of of [{[$PhBP^{iPr}_3$]Fe}$_2$(μ-N_2)][Na(THF)$_6$]·THF (**7**)

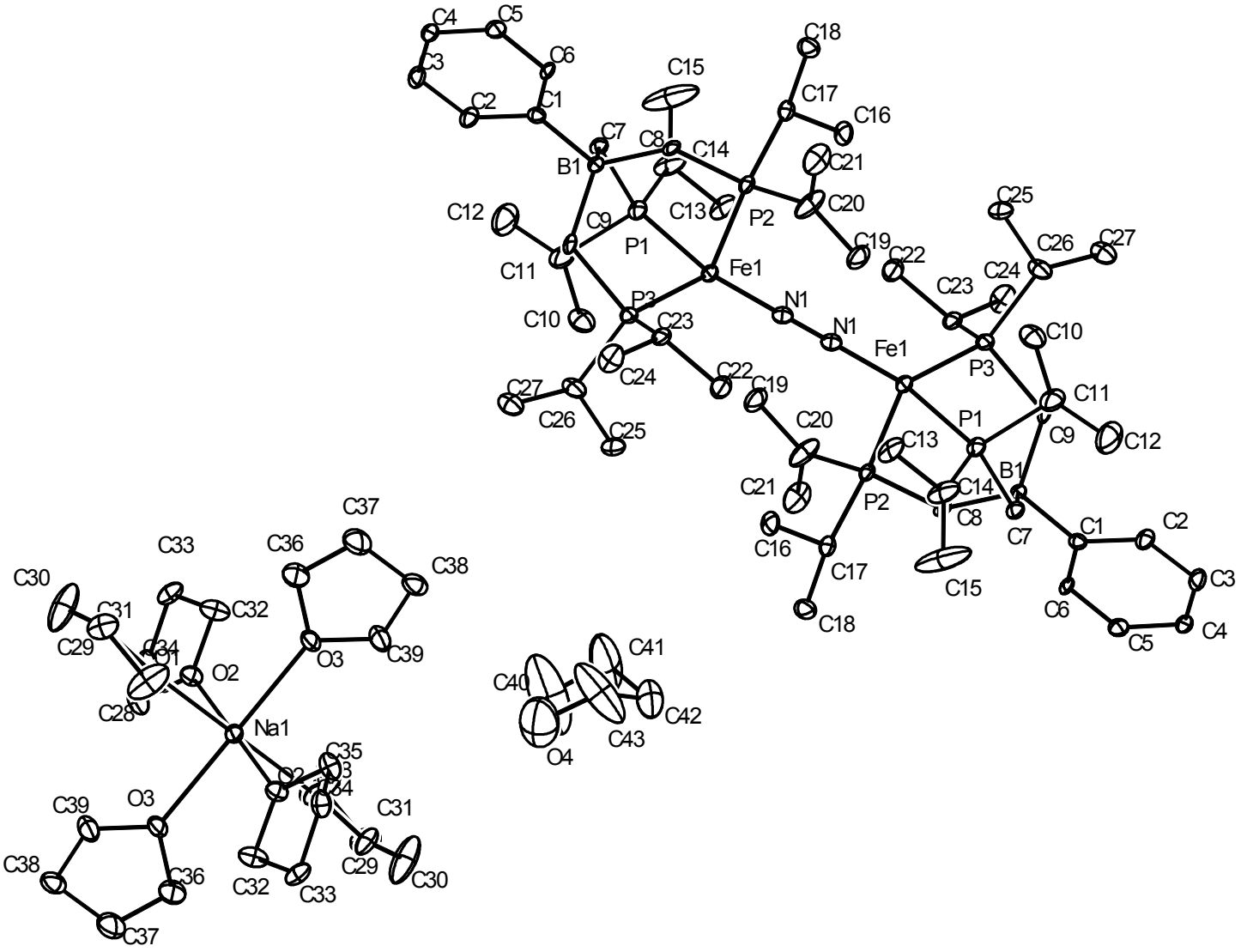

Table 1. Crystal data and structure refinement for $[\{(PhBP^{iPr}_3)Fe\}_2(\mu^2\text{-}N_2)][Na(THF)_6]\cdot THF$.

Identification code	tab38	
Empirical formula	$C_{43}H_{85}BFeNNa_{0.50}O_4P_3$	
Formula weight	851.18	
Temperature	96(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 11.7243(11) Å	α= 80.981(2)°.
	b = 14.5838(14) Å	β= 85.696(2)°.
	c = 15.0066(14) Å	γ = 71.417(2)°.
Volume	2401.2(4) $Å^3$	
Z	2	
Density (calculated)	1.177 Mg/m^3	
Absorption coefficient	0.457 mm^{-1}	
F(000)	927	
Crystal size	0.23 x 0.23 x 0.26 mm^3	
Theta range for data collection	1.49 to 28.54°.	
Index ranges	-15<=h<=15, -19<=k<=19, -20<=l<=20	
Reflections collected	50209	
Independent reflections	11199 [R(int) = 0.0951]	
Completeness to theta = 28.54°	91.6 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	11199 / 0 / 517	
Goodness-of-fit on F^2	1.553	
Final R indices [I>2sigma(I)]	R1 = 0.0609, wR2 = 0.1131	
R indices (all data)	R1 = 0.1338, wR2 = 0.1240	
Largest diff. peak and hole	0.970 and -0.883 e.$Å^{-3}$	

Special Refinement Details

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

One half of the $\{[PhBP^{iPr}_3]FeN\}^-$ and one half of the Na cation are generated by a symmetry element. The THF solvent molecule suffers from disorder in the atomic positions. Carbons 12 and 15 were disordered in two positions and split during the refinement. The population of the two positions were refined as a free variables during the refinement.

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [{(PhBP$^{iPr}_3$)Fe}$_2$(μ^2-N_2)][Na(THF)$_6$]·THF. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Fe(1)	8563(1)	6293(1)	5679(1)	24(1)
P(1)	8827(1)	7803(1)	5495(1)	26(1)
P(2)	8747(1)	5972(1)	7206(1)	22(1)
P(3)	6503(1)	6800(1)	5664(1)	26(1)
N(1)	9649(2)	5323(2)	5157(2)	23(1)
B(1)	6979(3)	7965(3)	7000(3)	22(1)
C(1)	6159(3)	8824(2)	7572(2)	21(1)
C(2)	5621(3)	9769(2)	7148(2)	29(1)
C(3)	4908(3)	10515(3)	7601(2)	32(1)
C(4)	4713(3)	10349(3)	8513(2)	31(1)
C(5)	5219(3)	9418(3)	8966(2)	32(1)
C(6)	5914(3)	8675(2)	8498(2)	24(1)
C(7)	8077(3)	8367(2)	6478(2)	25(1)
C(8)	7567(3)	6939(2)	7691(2)	22(1)
C(9)	6015(3)	7840(2)	6298(2)	24(1)
C(10)	8366(4)	8290(3)	3642(2)	47(1)
C(11)	8028(4)	8675(3)	4538(3)	48(1)
C(12A)	8050(9)	9742(5)	4463(5)	47(2)
C(12B)	7370(20)	9579(17)	4486(13)	66(7)
C(13)	11174(3)	7219(3)	4794(3)	47(1)
C(14)	10355(3)	7947(3)	5361(3)	56(1)
C(15A)	10933(7)	8103(8)	6105(6)	55(3)
C(15B)	10656(13)	8683(13)	5628(14)	35(5)
C(16)	11270(3)	5389(3)	7196(3)	40(1)
C(17)	10159(3)	6027(2)	7654(2)	28(1)
C(18)	10310(3)	5860(3)	8680(2)	39(1)
C(19)	9081(4)	3984(3)	7278(3)	57(1)
C(20)	8599(4)	4790(3)	7791(3)	56(1)
C(21)	8280(4)	4669(3)	8746(3)	62(1)
C(22)	6261(3)	4937(3)	5938(3)	44(1)
C(23)	5786(3)	5916(3)	6284(3)	34(1)
C(24)	4414(3)	6224(3)	6385(3)	58(1)
C(25)	6219(4)	6606(3)	3863(2)	48(1)
C(26)	5766(3)	7275(3)	4554(2)	50(1)
C(27)	4451(3)	7822(3)	4542(3)	47(1)
Na(1)	0	10000	0	27(1)
O(1)	1380(2)	8952(2)	-908(2)	39(1)
C(28)	2303(5)	9235(4)	-1428(4)	93(2)
C(29)	2867(4)	8502(3)	-2028(3)	59(1)
C(30)	2569(6)	7646(4)	-1584(4)	120(3)
C(31)	1493(4)	7978(3)	-1031(3)	51(1)
O(2)	-894(2)	8774(2)	384(2)	36(1)
C(32)	-1823(4)	8640(3)	-109(3)	48(1)
C(33)	-2112(4)	7749(3)	392(3)	46(1)
C(34)	-1698(3)	7691(3)	1337(3)	41(1)
C(35)	-608(3)	8043(3)	1155(3)	44(1)
O(3)	1262(2)	9413(2)	1266(2)	45(1)
C(36)	1129(5)	9790(4)	2098(3)	96(2)
C(37)	1825(5)	9081(4)	2753(3)	79(2)
C(38)	2609(4)	8271(3)	2279(3)	55(1)

C(39)	2365(4)	8634(3)	1298(3)	55(1)
O(4)	4213(5)	6361(4)	671(5)	184(2)
C(40)	5206(7)	6684(7)	429(9)	304(9)
C(41)	6168(7)	6038(5)	761(8)	209(5)
C(42)	6006(7)	5144(5)	967(6)	152(3)
C(43)	4761(7)	5593(8)	1362(5)	213(6)

Table 3. Bond lengths [Å] and angles [°] for [{(PhBP$^{iPr}_3$)Fe}$_2$(μ^2-N_2)][Na(THF)$_6$]·THF.

Fe(1)-N(1)	1.813(2)	C(14)-H(14)	1.0000
Fe(1)-P(2)	2.2780(10)	C(15A)-H(15A)	0.9800
Fe(1)-P(3)	2.2898(10)	C(15A)-H(15B)	0.9800
Fe(1)-P(1)	2.2922(11)	C(15A)-H(15C)	0.9800
P(1)-C(7)	1.830(3)	C(15B)-H(15D)	0.9800
P(1)-C(11)	1.851(4)	C(15B)-H(15E)	0.9800
P(1)-C(14)	1.862(4)	C(15B)-H(15F)	0.9800
P(2)-C(8)	1.833(3)	C(16)-C(17)	1.526(4)
P(2)-C(20)	1.862(4)	C(16)-H(16A)	0.9800
P(2)-C(17)	1.862(3)	C(16)-H(16B)	0.9800
P(3)-C(9)	1.831(3)	C(16)-H(16C)	0.9800
P(3)-C(23)	1.851(4)	C(17)-C(18)	1.536(4)
P(3)-C(26)	1.865(4)	C(17)-H(17)	1.0000
N(1)-N(1)#1	1.171(4)	C(18)-H(18A)	0.9800
B(1)-C(1)	1.636(5)	C(18)-H(18B)	0.9800
B(1)-C(8)	1.659(5)	C(18)-H(18C)	0.9800
B(1)-C(7)	1.674(5)	C(19)-C(20)	1.446(5)
B(1)-C(9)	1.670(5)	C(19)-H(19A)	0.9800
C(1)-C(2)	1.390(4)	C(19)-H(19B)	0.9800
C(1)-C(6)	1.396(4)	C(19)-H(19C)	0.9800
C(2)-C(3)	1.383(4)	C(20)-C(21)	1.451(5)
C(2)-H(2)	0.9500	C(20)-H(20)	1.0000
C(3)-C(4)	1.367(5)	C(21)-H(21A)	0.9800
C(3)-H(3)	0.9500	C(21)-H(21B)	0.9800
C(4)-C(5)	1.384(5)	C(21)-H(21C)	0.9800
C(4)-H(4)	0.9500	C(22)-C(23)	1.518(4)
C(5)-C(6)	1.387(4)	C(22)-H(22A)	0.9800
C(5)-H(5)	0.9500	C(22)-H(22B)	0.9800
C(6)-H(6)	0.9500	C(22)-H(22C)	0.9800
C(7)-H(7A)	0.9900	C(23)-C(24)	1.529(5)
C(7)-H(7B)	0.9900	C(23)-H(23)	1.0000
C(8)-H(8A)	0.9900	C(24)-H(24A)	0.9800
C(8)-H(8B)	0.9900	C(24)-H(24B)	0.9800
C(9)-H(9A)	0.9900	C(24)-H(24C)	0.9800
C(9)-H(9B)	0.9900	C(25)-C(26)	1.487(5)
C(10)-C(11)	1.515(5)	C(25)-H(25A)	0.9800
C(10)-H(10A)	0.9800	C(25)-H(25B)	0.9800
C(10)-H(10B)	0.9800	C(25)-H(25C)	0.9800
C(10)-H(10C)	0.9800	C(26)-C(27)	1.493(5)
C(11)-C(12B)	1.29(2)	C(26)-H(26)	1.0000
C(11)-C(12A)	1.550(8)	C(27)-H(27A)	0.9800
C(11)-H(11)	1.0000	C(27)-H(27B)	0.9800
C(12A)-H(12A)	0.9800	C(27)-H(27C)	0.9800
C(12A)-H(12B)	0.9800	Na(1)-O(2)#2	2.326(2)
C(12A)-H(12C)	0.9800	Na(1)-O(2)	2.326(2)
C(12B)-H(12D)	0.9800	Na(1)-O(1)	2.357(2)
C(12B)-H(12E)	0.9800	Na(1)-O(1)#2	2.357(2)
C(12B)-H(12F)	0.9800	Na(1)-O(3)	2.374(2)
C(13)-C(14)	1.511(5)	Na(1)-O(3)#2	2.374(2)
C(13)-H(13A)	0.9800	O(1)-C(28)	1.417(4)
C(13)-H(13B)	0.9800	O(1)-C(31)	1.423(4)
C(13)-H(13C)	0.9800	C(28)-C(29)	1.467(5)
C(14)-C(15B)	1.355(14)	C(28)-H(28A)	0.9900
C(14)-C(15A)	1.432(8)	C(28)-H(28B)	0.9900

C(29)-C(30)	1.451(6)	C(7)-P(1)-Fe(1)	105.54(11)
C(29)-H(29A)	0.9900	C(11)-P(1)-Fe(1)	115.22(14)
C(29)-H(29B)	0.9900	C(14)-P(1)-Fe(1)	121.49(15)
C(30)-C(31)	1.448(6)	C(8)-P(2)-C(20)	106.54(16)
C(30)-H(30A)	0.9900	C(8)-P(2)-C(17)	103.55(15)
C(30)-H(30B)	0.9900	C(20)-P(2)-C(17)	103.7(2)
C(31)-H(31A)	0.9900	C(8)-P(2)-Fe(1)	107.29(11)
C(31)-H(31B)	0.9900	C(20)-P(2)-Fe(1)	118.00(14)
O(2)-C(35)	1.424(4)	C(17)-P(2)-Fe(1)	116.51(11)
O(2)-C(32)	1.439(4)	C(9)-P(3)-C(23)	104.19(15)
C(32)-C(33)	1.518(5)	C(9)-P(3)-C(26)	104.70(16)
C(32)-H(32A)	0.9900	C(23)-P(3)-C(26)	107.96(19)
C(32)-H(32B)	0.9900	C(9)-P(3)-Fe(1)	105.38(11)
C(33)-C(34)	1.514(5)	C(23)-P(3)-Fe(1)	115.06(12)
C(33)-H(33A)	0.9900	C(26)-P(3)-Fe(1)	118.06(13)
C(33)-H(33B)	0.9900	N(1)#1-N(1)-Fe(1)	178.0(3)
C(34)-C(35)	1.514(5)	C(1)-B(1)-C(8)	110.3(3)
C(34)-H(34A)	0.9900	C(1)-B(1)-C(7)	106.4(3)
C(34)-H(34B)	0.9900	C(8)-B(1)-C(7)	110.0(3)
C(35)-H(35A)	0.9900	C(1)-B(1)-C(9)	104.2(3)
C(35)-H(35B)	0.9900	C(8)-B(1)-C(9)	111.7(3)
O(3)-C(36)	1.421(4)	C(7)-B(1)-C(9)	113.9(3)
O(3)-C(39)	1.421(4)	C(2)-C(1)-C(6)	114.8(3)
C(36)-C(37)	1.401(6)	C(2)-C(1)-B(1)	121.3(3)
C(36)-H(36A)	0.9900	C(6)-C(1)-B(1)	123.9(3)
C(36)-H(36B)	0.9900	C(3)-C(2)-C(1)	123.3(3)
C(37)-C(38)	1.490(6)	C(3)-C(2)-H(2)	118.3
C(37)-H(37A)	0.9900	C(1)-C(2)-H(2)	118.3
C(37)-H(37B)	0.9900	C(4)-C(3)-C(2)	120.3(3)
C(38)-C(39)	1.501(5)	C(4)-C(3)-H(3)	119.9
C(38)-H(38A)	0.9900	C(2)-C(3)-H(3)	119.9
C(38)-H(38B)	0.9900	C(3)-C(4)-C(5)	118.7(3)
C(39)-H(39A)	0.9900	C(3)-C(4)-H(4)	120.7
C(39)-H(39B)	0.9900	C(5)-C(4)-H(4)	120.7
O(4)-C(40)	1.393(7)	C(4)-C(5)-C(6)	120.2(3)
O(4)-C(43)	1.421(7)	C(4)-C(5)-H(5)	119.9
C(40)-C(41)	1.291(9)	C(6)-C(5)-H(5)	119.9
C(40)-H(40A)	0.9900	C(5)-C(6)-C(1)	122.6(3)
C(40)-H(40B)	0.9900	C(5)-C(6)-H(6)	118.7
C(41)-C(42)	1.362(8)	C(1)-C(6)-H(6)	118.7
C(41)-H(41A)	0.9900	B(1)-C(7)-P(1)	117.2(2)
C(41)-H(41B)	0.9900	B(1)-C(7)-H(7A)	108.0
C(42)-C(43)	1.515(9)	P(1)-C(7)-H(7A)	108.0
C(42)-H(42A)	0.9900	B(1)-C(7)-H(7B)	108.0
C(42)-H(42B)	0.9900	P(1)-C(7)-H(7B)	108.0
C(43)-H(43A)	0.9900	H(7A)-C(7)-H(7B)	107.2
C(43)-H(43B)	0.9900	B(1)-C(8)-P(2)	116.3(2)
N(1)-Fe(1)-P(2)	109.66(9)	B(1)-C(8)-H(8A)	108.2
N(1)-Fe(1)-P(3)	129.83(9)	P(2)-C(8)-H(8A)	108.2
P(2)-Fe(1)-P(3)	97.22(4)	B(1)-C(8)-H(8B)	108.2
N(1)-Fe(1)-P(1)	118.88(9)	P(2)-C(8)-H(8B)	108.2
P(2)-Fe(1)-P(1)	96.93(4)	H(8A)-C(8)-H(8B)	107.4
P(3)-Fe(1)-P(1)	97.96(4)	B(1)-C(9)-P(3)	118.5(2)
C(7)-P(1)-C(11)	102.83(17)	B(1)-C(9)-H(9A)	107.7
C(7)-P(1)-C(14)	108.05(16)	P(3)-C(9)-H(9A)	107.7
C(11)-P(1)-C(14)	102.1(2)	B(1)-C(9)-H(9B)	107.7

P(3)-C(9)-H(9B)	107.7	H(16A)-C(16)-H(16C)	109.5
H(9A)-C(9)-H(9B)	107.1	H(16B)-C(16)-H(16C)	109.5
C(11)-C(10)-H(10A)	109.5	C(16)-C(17)-C(18)	110.3(3)
C(11)-C(10)-H(10B)	109.5	C(16)-C(17)-P(2)	111.3(2)
H(10A)-C(10)-H(10B)	109.5	C(18)-C(17)-P(2)	118.9(2)
C(11)-C(10)-H(10C)	109.5	C(16)-C(17)-H(17)	105.0
H(10A)-C(10)-H(10C)	109.5	C(18)-C(17)-H(17)	105.0
H(10B)-C(10)-H(10C)	109.5	P(2)-C(17)-H(17)	105.0
C(12B)-C(11)-C(10)	115.1(9)	C(17)-C(18)-H(18A)	109.5
C(12B)-C(11)-C(12A)	35.4(11)	C(17)-C(18)-H(18B)	109.5
C(10)-C(11)-C(12A)	111.0(4)	H(18A)-C(18)-H(18B)	109.5
C(12B)-C(11)-P(1)	131.4(9)	C(17)-C(18)-H(18C)	109.5
C(10)-C(11)-P(1)	112.8(3)	H(18A)-C(18)-H(18C)	109.5
C(12A)-C(11)-P(1)	116.8(4)	H(18B)-C(18)-H(18C)	109.5
C(12B)-C(11)-H(11)	69.9	C(20)-C(19)-H(19A)	109.5
C(10)-C(11)-H(11)	105.0	C(20)-C(19)-H(19B)	109.5
C(12A)-C(11)-H(11)	105.0	H(19A)-C(19)-H(19B)	109.5
P(1)-C(11)-H(11)	105.0	C(20)-C(19)-H(19C)	109.5
C(11)-C(12A)-H(12A)	109.5	H(19A)-C(19)-H(19C)	109.5
C(11)-C(12A)-H(12B)	109.5	H(19B)-C(19)-H(19C)	109.5
C(11)-C(12A)-H(12C)	109.5	C(21)-C(20)-C(19)	123.6(4)
C(11)-C(12B)-H(12D)	109.5	C(21)-C(20)-P(2)	120.9(3)
C(11)-C(12B)-H(12E)	109.5	C(19)-C(20)-P(2)	114.1(3)
H(12D)-C(12B)-H(12E)	109.5	C(21)-C(20)-H(20)	94.0
C(11)-C(12B)-H(12F)	109.5	C(19)-C(20)-H(20)	94.0
H(12D)-C(12B)-H(12F)	109.5	P(2)-C(20)-H(20)	94.0
H(12E)-C(12B)-H(12F)	109.5	C(20)-C(21)-H(21A)	109.5
C(14)-C(13)-H(13A)	109.5	C(20)-C(21)-H(21B)	109.5
C(14)-C(13)-H(13B)	109.5	H(21A)-C(21)-H(21B)	109.5
H(13A)-C(13)-H(13B)	109.5	C(20)-C(21)-H(21C)	109.5
C(14)-C(13)-H(13C)	109.5	H(21A)-C(21)-H(21C)	109.5
H(13A)-C(13)-H(13C)	109.5	H(21B)-C(21)-H(21C)	109.5
H(13B)-C(13)-H(13C)	109.5	C(23)-C(22)-H(22A)	109.5
C(15B)-C(14)-C(15A)	42.0(6)	C(23)-C(22)-H(22B)	109.5
C(15B)-C(14)-C(13)	123.7(7)	H(22A)-C(22)-H(22B)	109.5
C(15A)-C(14)-C(13)	114.0(5)	C(23)-C(22)-H(22C)	109.5
C(15B)-C(14)-P(1)	124.8(7)	H(22A)-C(22)-H(22C)	109.5
C(15A)-C(14)-P(1)	121.2(4)	H(22B)-C(22)-H(22C)	109.5
C(13)-C(14)-P(1)	111.0(3)	C(22)-C(23)-C(24)	110.3(3)
C(15B)-C(14)-H(14)	60.7	C(22)-C(23)-P(3)	111.1(2)
C(15A)-C(14)-H(14)	102.6	C(24)-C(23)-P(3)	118.9(3)
C(13)-C(14)-H(14)	102.6	C(22)-C(23)-H(23)	105.1
P(1)-C(14)-H(14)	102.6	C(24)-C(23)-H(23)	105.1
C(14)-C(15A)-H(15A)	109.5	P(3)-C(23)-H(23)	105.1
C(14)-C(15A)-H(15B)	109.5	C(23)-C(24)-H(24A)	109.5
C(14)-C(15A)-H(15C)	109.5	C(23)-C(24)-H(24B)	109.5
C(14)-C(15B)-H(15D)	109.5	H(24A)-C(24)-H(24B)	109.5
C(14)-C(15B)-H(15E)	109.5	C(23)-C(24)-H(24C)	109.5
H(15D)-C(15B)-H(15E)	109.5	H(24A)-C(24)-H(24C)	109.5
C(14)-C(15B)-H(15F)	109.5	H(24B)-C(24)-H(24C)	109.5
H(15D)-C(15B)-H(15F)	109.5	C(26)-C(25)-H(25A)	109.5
H(15E)-C(15B)-H(15F)	109.5	C(26)-C(25)-H(25B)	109.5
C(17)-C(16)-H(16A)	109.5	H(25A)-C(25)-H(25B)	109.5
C(17)-C(16)-H(16B)	109.5	C(26)-C(25)-H(25C)	109.5
H(16A)-C(16)-H(16B)	109.5	H(25A)-C(25)-H(25C)	109.5
C(17)-C(16)-H(16C)	109.5	H(25B)-C(25)-H(25C)	109.5

C(25)-C(26)-C(27)	116.0(3)	C(32)-O(2)-Na(1)	125.6(2)
C(25)-C(26)-P(3)	113.8(3)	O(2)-C(32)-C(33)	106.6(3)
C(27)-C(26)-P(3)	118.7(3)	O(2)-C(32)-H(32A)	110.4
C(25)-C(26)-H(26)	101.4	C(33)-C(32)-H(32A)	110.4
C(27)-C(26)-H(26)	101.4	O(2)-C(32)-H(32B)	110.4
P(3)-C(26)-H(26)	101.4	C(33)-C(32)-H(32B)	110.4
C(26)-C(27)-H(27A)	109.5	H(32A)-C(32)-H(32B)	108.6
C(26)-C(27)-H(27B)	109.5	C(34)-C(33)-C(32)	102.8(3)
H(27A)-C(27)-H(27B)	109.5	C(34)-C(33)-H(33A)	111.2
C(26)-C(27)-H(27C)	109.5	C(32)-C(33)-H(33A)	111.2
H(27A)-C(27)-H(27C)	109.5	C(34)-C(33)-H(33B)	111.2
H(27B)-C(27)-H(27C)	109.5	C(32)-C(33)-H(33B)	111.2
O(2)#2-Na(1)-O(2)	180.000(1)	H(33A)-C(33)-H(33B)	109.1
O(2)#2-Na(1)-O(1)	92.74(8)	C(33)-C(34)-C(35)	102.0(3)
O(2)-Na(1)-O(1)	87.26(8)	C(33)-C(34)-H(34A)	111.4
O(2)#2-Na(1)-O(1)#2	87.26(8)	C(35)-C(34)-H(34A)	111.4
O(2)-Na(1)-O(1)#2	92.74(8)	C(33)-C(34)-H(34B)	111.4
O(1)-Na(1)-O(1)#2	180.0	C(35)-C(34)-H(34B)	111.4
O(2)#2-Na(1)-O(3)	88.19(9)	H(34A)-C(34)-H(34B)	109.2
O(2)-Na(1)-O(3)	91.81(9)	O(2)-C(35)-C(34)	105.4(3)
O(1)-Na(1)-O(3)	92.49(9)	O(2)-C(35)-H(35A)	110.7
O(1)#2-Na(1)-O(3)	87.51(9)	C(34)-C(35)-H(35A)	110.7
O(2)#2-Na(1)-O(3)#2	91.81(9)	O(2)-C(35)-H(35B)	110.7
O(2)-Na(1)-O(3)#2	88.19(9)	C(34)-C(35)-H(35B)	110.7
O(1)-Na(1)-O(3)#2	87.51(9)	H(35A)-C(35)-H(35B)	108.8
O(1)#2-Na(1)-O(3)#2	92.49(9)	C(36)-O(3)-C(39)	106.7(3)
O(3)-Na(1)-O(3)#2	180.00(13)	C(36)-O(3)-Na(1)	128.2(3)
C(28)-O(1)-C(31)	108.3(3)	C(39)-O(3)-Na(1)	125.0(2)
C(28)-O(1)-Na(1)	121.8(2)	C(37)-C(36)-O(3)	109.7(4)
C(31)-O(1)-Na(1)	129.9(2)	C(37)-C(36)-H(36A)	109.7
O(1)-C(28)-C(29)	108.4(4)	O(3)-C(36)-H(36A)	109.7
O(1)-C(28)-H(28A)	110.0	C(37)-C(36)-H(36B)	109.7
C(29)-C(28)-H(28A)	110.0	O(3)-C(36)-H(36B)	109.7
O(1)-C(28)-H(28B)	110.0	H(36A)-C(36)-H(36B)	108.2
C(29)-C(28)-H(28B)	110.0	C(36)-C(37)-C(38)	107.3(4)
H(28A)-C(28)-H(28B)	108.4	C(36)-C(37)-H(37A)	110.2
C(30)-C(29)-C(28)	103.0(4)	C(38)-C(37)-H(37A)	110.2
C(30)-C(29)-H(29A)	111.2	C(36)-C(37)-H(37B)	110.2
C(28)-C(29)-H(29A)	111.2	C(38)-C(37)-H(37B)	110.2
C(30)-C(29)-H(29B)	111.2	H(37A)-C(37)-H(37B)	108.5
C(28)-C(29)-H(29B)	111.2	C(37)-C(38)-C(39)	104.1(3)
H(29A)-C(29)-H(29B)	109.1	C(37)-C(38)-H(38A)	110.9
C(31)-C(30)-C(29)	107.7(4)	C(39)-C(38)-H(38A)	110.9
C(31)-C(30)-H(30A)	110.2	C(37)-C(38)-H(38B)	110.9
C(29)-C(30)-H(30A)	110.2	C(39)-C(38)-H(38B)	110.9
C(31)-C(30)-H(30B)	110.2	H(38A)-C(38)-H(38B)	108.9
C(29)-C(30)-H(30B)	110.2	O(3)-C(39)-C(38)	106.3(3)
H(30A)-C(30)-H(30B)	108.5	O(3)-C(39)-H(39A)	110.5
O(1)-C(31)-C(30)	106.6(4)	C(38)-C(39)-H(39A)	110.5
O(1)-C(31)-H(31A)	110.4	O(3)-C(39)-H(39B)	110.5
C(30)-C(31)-H(31A)	110.4	C(38)-C(39)-H(39B)	110.5
O(1)-C(31)-H(31B)	110.4	H(39A)-C(39)-H(39B)	108.7
C(30)-C(31)-H(31B)	110.4	C(40)-O(4)-C(43)	97.6(6)
H(31A)-C(31)-H(31B)	108.6	C(41)-C(40)-O(4)	109.9(7)
C(35)-O(2)-C(32)	109.6(3)	C(41)-C(40)-H(40A)	109.6
C(35)-O(2)-Na(1)	124.8(2)	O(4)-C(40)-H(40A)	109.7

C(41)-C(40)-H(40B)	109.8	C(43)-C(42)-H(42A)	113.5
O(4)-C(40)-H(40B)	109.7	C(41)-C(42)-H(42B)	113.4
H(40A)-C(40)-H(40B)	108.2	C(43)-C(42)-H(42B)	113.4
C(40)-C(41)-C(42)	111.0(8)	H(42A)-C(42)-H(42B)	110.8
C(40)-C(41)-H(41A)	109.4	O(4)-C(43)-C(42)	103.0(5)
C(42)-C(41)-H(41A)	109.4	O(4)-C(43)-H(43A)	111.2
C(40)-C(41)-H(41B)	109.5	C(42)-C(43)-H(43A)	111.2
C(42)-C(41)-H(41B)	109.5	O(4)-C(43)-H(43B)	111.1
H(41A)-C(41)-H(41B)	108.0	C(42)-C(43)-H(43B)	111.1
C(41)-C(42)-C(43)	91.1(7)	H(43A)-C(43)-H(43B)	109.1
C(41)-C(42)-H(42A)	113.5		

Symmetry transformations used to generate equivalent atoms:
#1 -x+2,-y+1,-z+1 #2 -x,-y+2,-z

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for [{$(PhBP^{iPr}_3)Fe$}$_2(\mu^2\text{-}N_2)$][$Na(THF)_6$]·THF. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^2U^{11} + ...\ + 2\ h\ k\ a\ b\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Fe(1)	24(1)	24(1)	21(1)	-11(1)	4(1)	0(1)
P(1)	27(1)	28(1)	22(1)	-8(1)	7(1)	-5(1)
P(2)	24(1)	19(1)	23(1)	-7(1)	4(1)	-3(1)
P(3)	24(1)	29(1)	23(1)	-14(1)	0(1)	-3(1)
N(1)	25(2)	24(2)	20(2)	-7(1)	5(1)	-8(1)
B(1)	22(2)	22(2)	20(2)	-10(2)	3(2)	-2(2)
C(1)	18(2)	26(2)	23(2)	-10(2)	3(2)	-8(2)
C(2)	34(2)	26(2)	24(2)	-5(2)	6(2)	-4(2)
C(3)	28(2)	26(2)	35(2)	-11(2)	2(2)	1(2)
C(4)	25(2)	30(2)	34(2)	-20(2)	1(2)	4(2)
C(5)	33(2)	40(2)	21(2)	-14(2)	1(2)	-4(2)
C(6)	24(2)	22(2)	23(2)	-8(2)	0(2)	0(2)
C(7)	26(2)	22(2)	24(2)	-8(2)	5(2)	-3(2)
C(8)	20(2)	25(2)	21(2)	-12(2)	4(2)	-5(2)
C(9)	23(2)	24(2)	23(2)	-9(2)	7(2)	-2(2)
C(10)	49(3)	63(3)	27(2)	-8(2)	6(2)	-15(2)
C(11)	64(3)	41(3)	30(2)	-4(2)	5(2)	-5(2)
C(12A)	59(6)	35(4)	46(4)	11(3)	-13(4)	-19(4)
C(12B)	59(15)	97(17)	48(11)	-19(11)	9(10)	-30(13)
C(13)	30(2)	57(3)	62(3)	-34(2)	19(2)	-17(2)
C(14)	35(3)	70(3)	78(3)	-50(3)	23(2)	-25(2)
C(15A)	47(5)	81(7)	50(5)	-28(5)	18(4)	-35(5)
C(15B)	31(8)	27(9)	45(11)	0(8)	-5(7)	-6(7)
C(16)	27(2)	40(2)	45(2)	-5(2)	3(2)	-2(2)
C(17)	25(2)	24(2)	29(2)	-4(2)	1(2)	-2(2)
C(18)	29(2)	49(3)	37(2)	-13(2)	-9(2)	-6(2)
C(19)	95(4)	25(2)	48(3)	-3(2)	-9(3)	-14(2)
C(20)	96(4)	32(2)	43(3)	-15(2)	36(3)	-29(2)
C(21)	98(4)	46(3)	46(3)	-4(2)	18(3)	-32(3)
C(22)	38(2)	34(2)	59(3)	-13(2)	-3(2)	-9(2)
C(23)	28(2)	36(2)	42(2)	-16(2)	-3(2)	-9(2)
C(24)	41(3)	42(3)	95(4)	-21(3)	19(3)	-18(2)
C(25)	68(3)	49(3)	28(2)	-15(2)	-11(2)	-12(2)
C(26)	39(3)	69(3)	30(2)	-22(2)	-9(2)	10(2)
C(27)	33(2)	73(3)	32(2)	-6(2)	-3(2)	-13(2)
Na(1)	27(1)	27(1)	25(1)	-4(1)	-1(1)	-4(1)
O(1)	38(2)	37(2)	48(2)	-20(1)	16(1)	-17(1)
C(28)	87(4)	99(4)	126(5)	-73(4)	82(4)	-65(4)
C(29)	45(3)	87(4)	57(3)	-31(3)	20(2)	-33(3)
C(30)	156(6)	54(4)	113(5)	-16(3)	91(5)	-2(4)
C(31)	65(3)	33(2)	53(3)	-17(2)	19(2)	-13(2)
O(2)	39(2)	39(2)	32(2)	2(1)	-8(1)	-13(1)
C(32)	52(3)	68(3)	31(2)	-5(2)	-5(2)	-31(2)
C(33)	45(3)	44(3)	54(3)	-26(2)	14(2)	-15(2)
C(34)	37(2)	33(2)	46(3)	4(2)	6(2)	-8(2)
C(35)	38(2)	44(3)	42(3)	8(2)	-5(2)	-8(2)
O(3)	50(2)	47(2)	30(2)	-5(1)	-14(1)	-1(2)
C(36)	149(6)	76(4)	36(3)	-17(3)	-43(3)	15(4)
C(37)	78(4)	104(4)	42(3)	-3(3)	-16(3)	-10(3)
C(38)	70(3)	43(3)	54(3)	10(2)	-33(3)	-23(2)
C(39)	44(3)	58(3)	50(3)	0(2)	-11(2)	2(2)

O(4)	107(4)	146(5)	286(8)	-6(5)	-3(5)	-34(4)
C(40)	63(5)	202(10)	590(20)	229(12)	-39(9)	-83(6)
C(41)	96(6)	70(5)	457(17)	22(8)	-46(8)	-37(5)
C(42)	148(8)	70(5)	223(9)	-6(5)	-88(7)	-2(5)
C(43)	80(5)	393(15)	106(6)	166(8)	-19(4)	-80(7)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for [{(PhBP$^{iPr}_3$)Fe}$_2$(μ^2-N_2)][Na(THF)$_6$]·THF.

	x	y	z	U(eq)
H(2)	5750	9910	6516	35
H(3)	4551	11146	7277	38
H(4)	4239	10863	8830	37
H(5)	5090	9288	9599	38
H(6)	6235	8038	8821	29
H(7A)	8698	8274	6926	29
H(7B)	7735	9079	6285	29
H(8A)	6908	6675	7932	26
H(8B)	7905	7102	8209	26
H(9A)	5779	8443	5856	29
H(9B)	5281	7804	6654	29
H(10A)	7794	8701	3186	70
H(10B)	8342	7617	3706	70
H(10C)	9180	8303	3453	70
H(11)	7162	8718	4649	58
H(12A)	8861	9766	4279	70
H(12B)	7827	9974	5049	70
H(12C)	7475	10159	4012	70
H(12D)	7869	10002	4288	99
H(12E)	7001	9712	5079	99
H(12F)	6735	9709	4049	99
H(13A)	11959	7331	4706	71
H(13B)	10815	7300	4206	71
H(13C)	11280	6555	5102	71
H(14)	10213	8580	4949	67
H(15A)	11133	7512	6551	82
H(15B)	10389	8650	6385	82
H(15C)	11671	8254	5889	82
H(15D)	11513	8459	5767	53
H(15E)	10176	8885	6168	53
H(15F)	10496	9239	5143	53
H(16A)	11982	5550	7332	60
H(16B)	11166	5506	6541	60
H(16C)	11375	4699	7419	60
H(17)	10165	6713	7453	33
H(18A)	10478	5164	8907	58
H(18B)	9569	6237	8970	58
H(18C)	10981	6074	8819	58
H(19A)	9960	3746	7316	85
H(19B)	8859	4204	6645	85
H(19C)	8752	3455	7524	85
H(20)	7755	4935	7596	67
H(21A)	7786	4232	8863	94
H(21B)	7826	5307	8924	94
H(21C)	9015	4388	9096	94
H(22A)	6003	4447	6353	65
H(22B)	7143	4736	5899	65
H(22C)	5945	4994	5339	65
H(23)	6081	5790	6914	41
H(24A)	4068	6247	5805	87

H(24B)	4116	6872	6579	87
H(24C)	4178	5751	6838	87
H(25A)	5868	6071	3985	73
H(25B)	7098	6338	3886	73
H(25C)	5990	6968	3262	73
H(26)	6120	7809	4321	60
H(27A)	4248	8215	3950	70
H(27B)	4250	8251	5010	70
H(27C)	3991	7358	4658	70
H(28A)	2910	9278	-1026	112
H(28B)	1960	9884	-1788	112
H(29A)	2528	8714	-2639	71
H(29B)	3749	8374	-2077	71
H(30A)	2426	7274	-2039	144
H(30B)	3241	7214	-1205	144
H(31A)	779	7963	-1334	61
H(31B)	1564	7554	-441	61
H(32A)	-1538	8535	-733	57
H(32B)	-2547	9223	-133	57
H(33A)	-2986	7840	391	55
H(33B)	-1666	7154	122	55
H(34A)	-1478	7014	1655	49
H(34B)	-2326	8123	1696	49
H(35A)	-458	8320	1679	53
H(35B)	117	7499	1030	53
H(36A)	1390	10381	2018	115
H(36B)	272	9978	2299	115
H(37A)	1299	8836	3210	95
H(37B)	2324	9364	3061	95
H(38A)	2396	7664	2470	66
H(38B)	3468	8142	2403	66
H(39A)	3024	8869	1003	66
H(39B)	2296	8105	986	66
H(40A)	5294	6798	-237	365
H(40B)	5087	7309	659	365
H(41A)	6376	6236	1313	251
H(41B)	6845	5999	318	251
H(42A)	6575	4693	1416	182
H(42B)	6001	4828	430	182
H(43A)	4314	5112	1486	256
H(43B)	4804	5850	1927	256

Figure 2. Fully labeled drawing of of [{[PhBP$^{iPr}_3$]Co}$_2$(μ-N$_2$)][Na(THF)$_6$]·THF (**8**)

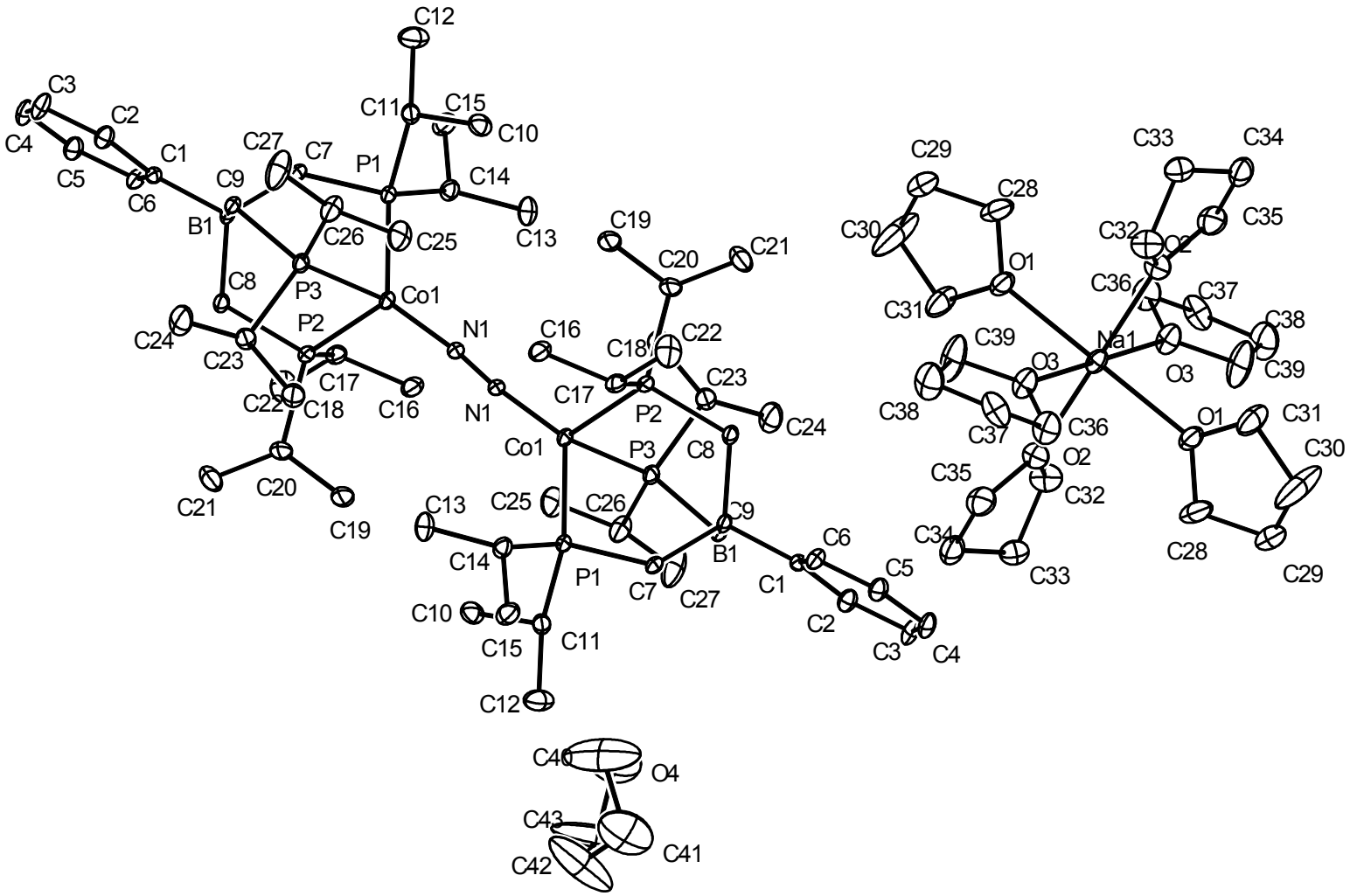

Table 6. Crystal data and structure refinement for [{(PhBP$^{iPr}_3$)Co}$_2$(μ^2-N$_2$)][Na(THF)$_6$]·THF.

Identification code	tab37	
Empirical formula	$C_{43}H_{85}BCoNNa_{0.50}O_4P_3$	
Formula weight	854.26	
Temperature	96(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 11.6846(10) Å	α= 80.204(2)°.
	b = 14.7047(13) Å	β= 85.159(2)°.
	c = 15.0700(13) Å	γ = 70.543(2)°.
Volume	2404.8(4) Å^3	
Z	2	
Density (calculated)	1.180 Mg/m^3	
Absorption coefficient	0.499 mm^{-1}	
F(000)	929	
Crystal size	0.19 x 0.21 x 0.34 mm^3	
Theta range for data collection	1.49 to 28.48°.	
Index ranges	-15<=h<=15, -19<=k<=19, -20<=l<=20	
Reflections collected	50199	
Independent reflections	11193 [R(int) = 0.0749]	
Completeness to theta = 28.48°	91.8 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	11193 / 0 / 507	
Goodness-of-fit on F^2	1.779	
Final R indices [I>2sigma(I)]	R1 = 0.0595, wR2 = 0.1162	
R indices (all data)	R1 = 0.1073, wR2 = 0.1235	
Largest diff. peak and hole	0.983 and -0.846 e.Å^{-3}	

Special Refinement Details

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of F^2 > 2σ(F^2) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

One half of the {[PhBP$^{iPr}_3$]CoN}$^-$ and one half of the Na cation are generated by a symmetry element. The THF solvent molecule suffers from disorder in the atomic positions. Carbon 21 was disordered in two positions and split during the refinement. The population of the two positions was refined as a free variable during the refinement.

Table 7. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $[\{(PhBP^{iPr}_3)Co\}_2(\mu^2\text{-}N_6)][Na(THF)_6]\cdot THF$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Co(1)	8504(1)	6304(1)	5603(1)	24(1)
P(1)	6477(1)	6851(1)	5645(1)	23(1)
P(2)	8708(1)	7776(1)	5483(1)	26(1)
P(3)	8629(1)	6021(1)	7111(1)	25(1)
N(1)	9617(2)	5298(2)	5150(2)	22(1)
B(1)	6856(3)	8006(3)	6960(2)	24(1)
C(1)	6049(3)	8861(2)	7547(2)	24(1)
C(2)	5856(3)	8713(2)	8470(2)	31(1)
C(3)	5203(3)	9460(3)	8949(2)	40(1)
C(4)	4707(3)	10396(2)	8509(2)	37(1)
C(5)	4850(3)	10565(2)	7590(2)	33(1)
C(6)	5503(3)	9818(2)	7126(2)	30(1)
C(7)	5897(3)	7902(2)	6249(2)	23(1)
C(8)	7962(3)	8392(2)	6433(2)	24(1)
C(9)	7445(3)	6964(2)	7634(2)	24(1)
C(10)	6267(3)	4974(2)	5951(2)	40(1)
C(11)	5764(3)	5964(2)	6269(2)	30(1)
C(12)	4363(3)	6266(3)	6336(3)	59(1)
C(13)	6148(3)	6567(3)	3883(2)	43(1)
C(14)	5738(3)	7307(2)	4532(2)	32(1)
C(15)	4380(3)	7828(3)	4520(2)	46(1)
C(16)	8291(3)	8213(3)	3623(2)	41(1)
C(17)	7920(3)	8633(2)	4502(2)	33(1)
C(18)	7953(4)	9679(3)	4372(3)	61(1)
C(19)	11134(3)	7167(3)	4831(3)	57(1)
C(20)	10275(3)	7852(3)	5379(3)	67(1)
C(21A)	10709(18)	8297(17)	5954(17)	56(5)
C(21B)	10574(17)	8677(14)	5625(16)	47(4)
C(22)	11178(3)	5367(3)	7115(3)	45(1)
C(23)	10068(3)	6069(2)	7519(2)	34(1)
C(24)	10222(4)	5968(4)	8538(3)	80(2)
C(25)	9135(4)	3988(2)	7254(3)	50(1)
C(26)	8485(4)	4843(2)	7707(2)	44(1)
C(27)	8308(5)	4688(3)	8694(3)	78(2)
Na(1)	10000	10000	10000	36(1)
O(1)	11436(2)	8920(2)	9134(2)	51(1)
C(28)	12352(5)	9172(4)	8599(4)	109(2)
C(29)	12848(5)	8444(4)	7982(4)	95(2)
C(30)	12487(7)	7661(4)	8344(4)	164(4)
C(31)	11523(4)	7948(3)	9036(3)	74(1)
O(2)	9078(2)	8797(2)	10368(2)	48(1)
C(32)	9351(4)	8043(3)	11110(3)	62(1)
C(33)	8320(3)	7638(3)	11255(3)	52(1)
C(34)	7897(4)	7791(3)	10306(3)	61(1)
C(35)	8124(4)	8721(3)	9871(3)	63(1)
O(3)	8803(2)	10615(2)	8701(2)	53(1)
C(36)	7675(4)	11391(3)	8656(3)	62(1)
C(37)	7422(4)	11721(3)	7678(3)	65(1)
C(38)	8129(4)	10833(4)	7258(3)	85(2)
C(39)	9032(5)	10279(4)	7852(3)	97(2)

O(4)	5753(4)	3668(4)	9372(4)	162(2)
C(40)	5225(7)	4437(8)	8665(5)	227(6)
C(41)	3883(6)	4782(7)	9051(6)	172(3)
C(42)	3844(10)	3915(7)	9080(9)	280(9)
C(43)	4774(8)	3335(7)	9654(8)	278(8)

Table 8. Bond lengths [Å] and angles [°] for [{(PhBP$^{iPr}_3$)Co}$_2$(μ^2-N$_2$)][Na(THF)$_6$]·THF.

Co(1)-N(1)	1.805(2)	C(16)-H(16A)	0.9800
Co(1)-P(2)	2.2300(9)	C(16)-H(16B)	0.9800
Co(1)-P(1)	2.2318(9)	C(16)-H(16C)	0.9800
Co(1)-P(3)	2.2487(9)	C(17)-C(18)	1.530(4)
P(1)-C(7)	1.830(3)	C(17)-H(17)	1.0000
P(1)-C(11)	1.859(3)	C(18)-H(18A)	0.9800
P(1)-C(14)	1.867(3)	C(18)-H(18B)	0.9800
P(2)-C(8)	1.826(3)	C(18)-H(18C)	0.9800
P(2)-C(17)	1.857(3)	C(19)-C(20)	1.475(5)
P(2)-C(20)	1.864(4)	C(19)-H(19A)	0.9800
P(3)-C(9)	1.834(3)	C(19)-H(19B)	0.9800
P(3)-C(23)	1.865(3)	C(19)-H(19C)	0.9800
P(3)-C(26)	1.864(3)	C(20)-C(21A)	1.386(18)
N(1)-N(1)#1	1.147(4)	C(20)-C(21B)	1.478(18)
B(1)-C(1)	1.643(4)	C(20)-H(20)	1.0000
B(1)-C(9)	1.656(5)	C(21A)-H(21A)	0.9800
B(1)-C(8)	1.669(5)	C(21A)-H(21B)	0.9800
B(1)-C(7)	1.674(4)	C(21A)-H(21C)	0.9800
C(1)-C(2)	1.382(4)	C(21B)-H(21D)	0.9800
C(1)-C(6)	1.398(4)	C(21B)-H(21E)	0.9800
C(2)-C(3)	1.389(4)	C(21B)-H(21F)	0.9800
C(2)-H(2)	0.9500	C(22)-C(23)	1.519(4)
C(3)-C(4)	1.376(5)	C(22)-H(22A)	0.9800
C(3)-H(3)	0.9500	C(22)-H(22B)	0.9800
C(4)-C(5)	1.370(4)	C(22)-H(22C)	0.9800
C(4)-H(4)	0.9500	C(23)-C(24)	1.538(5)
C(5)-C(6)	1.374(4)	C(23)-H(23)	1.0000
C(5)-H(5)	0.9500	C(24)-H(24A)	0.9800
C(6)-H(6)	0.9500	C(24)-H(24B)	0.9800
C(7)-H(7A)	0.9900	C(24)-H(24C)	0.9800
C(7)-H(7B)	0.9900	C(25)-C(26)	1.481(4)
C(8)-H(8A)	0.9900	C(25)-H(25A)	0.9800
C(8)-H(8B)	0.9900	C(25)-H(25B)	0.9800
C(9)-H(9A)	0.9900	C(25)-H(25C)	0.9800
C(9)-H(9B)	0.9900	C(26)-C(27)	1.473(5)
C(10)-C(11)	1.521(4)	C(26)-H(26)	1.0000
C(10)-H(10A)	0.9800	C(27)-H(27A)	0.9800
C(10)-H(10B)	0.9800	C(27)-H(27B)	0.9800
C(10)-H(10C)	0.9800	C(27)-H(27C)	0.9800
C(11)-C(12)	1.546(4)	Na(1)-O(2)#2	2.331(2)
C(11)-H(11)	1.0000	Na(1)-O(2)	2.331(2)
C(12)-H(12A)	0.9800	Na(1)-O(1)	2.367(2)
C(12)-H(12B)	0.9800	Na(1)-O(1)#2	2.367(2)
C(12)-H(12C)	0.9800	Na(1)-O(3)	2.372(2)
C(13)-C(14)	1.518(4)	Na(1)-O(3)#2	2.372(2)
C(13)-H(13A)	0.9800	O(1)-C(28)	1.397(5)
C(13)-H(13B)	0.9800	O(1)-C(31)	1.431(4)
C(13)-H(13C)	0.9800	C(28)-C(29)	1.477(6)
C(14)-C(15)	1.515(4)	C(28)-H(28A)	0.9900
C(14)-H(14)	1.0000	C(28)-H(28B)	0.9900
C(15)-H(15A)	0.9800	C(29)-C(30)	1.368(7)
C(15)-H(15B)	0.9800	C(29)-H(29A)	0.9900
C(15)-H(15C)	0.9800	C(29)-H(29B)	0.9900
C(16)-C(17)	1.526(4)	C(30)-C(31)	1.472(6)

C(30)-H(30A)	0.9900	C(8)-P(2)-C(20)	106.42(15)
C(30)-H(30B)	0.9900	C(17)-P(2)-C(20)	104.3(2)
C(31)-H(31A)	0.9900	C(8)-P(2)-Co(1)	111.32(10)
C(31)-H(31B)	0.9900	C(17)-P(2)-Co(1)	112.91(11)
O(2)-C(32)	1.408(4)	C(20)-P(2)-Co(1)	117.91(14)
O(2)-C(35)	1.438(4)	C(9)-P(3)-C(23)	103.63(14)
C(32)-C(33)	1.498(5)	C(9)-P(3)-C(26)	104.82(15)
C(32)-H(32A)	0.9900	C(23)-P(3)-C(26)	104.65(17)
C(32)-H(32B)	0.9900	C(9)-P(3)-Co(1)	111.27(10)
C(33)-C(34)	1.507(5)	C(23)-P(3)-Co(1)	113.89(11)
C(33)-H(33A)	0.9900	C(26)-P(3)-Co(1)	117.30(12)
C(33)-H(33B)	0.9900	N(1)#1-N(1)-Co(1)	174.6(3)
C(34)-C(35)	1.509(5)	C(1)-B(1)-C(9)	110.5(2)
C(34)-H(34A)	0.9900	C(1)-B(1)-C(8)	106.3(2)
C(34)-H(34B)	0.9900	C(9)-B(1)-C(8)	110.0(2)
C(35)-H(35A)	0.9900	C(1)-B(1)-C(7)	106.0(2)
C(35)-H(35B)	0.9900	C(9)-B(1)-C(7)	111.0(2)
O(3)-C(36)	1.425(4)	C(8)-B(1)-C(7)	112.9(2)
O(3)-C(39)	1.423(5)	C(2)-C(1)-C(6)	114.8(3)
C(36)-C(37)	1.492(5)	C(2)-C(1)-B(1)	124.3(3)
C(36)-H(36A)	0.9900	C(6)-C(1)-B(1)	120.9(3)
C(36)-H(36B)	0.9900	C(1)-C(2)-C(3)	122.7(3)
C(37)-C(38)	1.504(6)	C(1)-C(2)-H(2)	118.6
C(37)-H(37A)	0.9900	C(3)-C(2)-H(2)	118.6
C(37)-H(37B)	0.9900	C(4)-C(3)-C(2)	120.4(3)
C(38)-C(39)	1.384(5)	C(4)-C(3)-H(3)	119.8
C(38)-H(38A)	0.9900	C(2)-C(3)-H(3)	119.8
C(38)-H(38B)	0.9900	C(5)-C(4)-C(3)	118.4(3)
C(39)-H(39A)	0.9900	C(5)-C(4)-H(4)	120.8
C(39)-H(39B)	0.9900	C(3)-C(4)-H(4)	120.8
O(4)-C(43)	1.397(7)	C(4)-C(5)-C(6)	120.5(3)
O(4)-C(40)	1.424(7)	C(4)-C(5)-H(5)	119.8
C(40)-C(41)	1.573(9)	C(6)-C(5)-H(5)	119.8
C(40)-H(40A)	0.9900	C(5)-C(6)-C(1)	123.1(3)
C(40)-H(40B)	0.9900	C(5)-C(6)-H(6)	118.5
C(41)-C(42)	1.284(9)	C(1)-C(6)-H(6)	118.5
C(41)-H(41A)	0.9900	B(1)-C(7)-P(1)	115.60(19)
C(41)-H(41B)	0.9900	B(1)-C(7)-H(7A)	108.4
C(42)-C(43)	1.395(10)	P(1)-C(7)-H(7A)	108.4
C(42)-H(42A)	0.9900	B(1)-C(7)-H(7B)	108.4
C(42)-H(42B)	0.9900	P(1)-C(7)-H(7B)	108.4
C(43)-H(43A)	0.9900	H(7A)-C(7)-H(7B)	107.4
C(43)-H(43B)	0.9900	B(1)-C(8)-P(2)	115.13(19)
N(1)-Co(1)-P(2)	122.95(8)	B(1)-C(8)-H(8A)	108.5
N(1)-Co(1)-P(1)	133.30(8)	P(2)-C(8)-H(8A)	108.5
P(2)-Co(1)-P(1)	95.10(3)	B(1)-C(8)-H(8B)	108.5
N(1)-Co(1)-P(3)	108.39(8)	P(2)-C(8)-H(8B)	108.5
P(2)-Co(1)-P(3)	93.53(3)	H(8A)-C(8)-H(8B)	107.5
P(1)-Co(1)-P(3)	93.58(3)	B(1)-C(9)-P(3)	115.2(2)
C(7)-P(1)-C(11)	103.63(14)	B(1)-C(9)-H(9A)	108.5
C(7)-P(1)-C(14)	103.69(14)	P(3)-C(9)-H(9A)	108.5
C(11)-P(1)-C(14)	106.93(15)	B(1)-C(9)-H(9B)	108.5
C(7)-P(1)-Co(1)	111.00(10)	P(3)-C(9)-H(9B)	108.5
C(11)-P(1)-Co(1)	114.38(10)	H(9A)-C(9)-H(9B)	107.5
C(14)-P(1)-Co(1)	115.98(10)	C(11)-C(10)-H(10A)	109.5
C(8)-P(2)-C(17)	102.60(14)	C(11)-C(10)-H(10B)	109.5

H(10A)-C(10)-H(10B)	109.5	H(19A)-C(19)-H(19C)	109.5
C(11)-C(10)-H(10C)	109.5	H(19B)-C(19)-H(19C)	109.5
H(10A)-C(10)-H(10C)	109.5	C(21A)-C(20)-C(21B)	26.6(9)
H(10B)-C(10)-H(10C)	109.5	C(21A)-C(20)-C(19)	119.9(8)
C(10)-C(11)-C(12)	109.0(3)	C(21B)-C(20)-C(19)	120.8(8)
C(10)-C(11)-P(1)	112.1(2)	C(21A)-C(20)-P(2)	123.1(8)
C(12)-C(11)-P(1)	118.7(2)	C(21B)-C(20)-P(2)	122.9(8)
C(10)-C(11)-H(11)	105.3	C(19)-C(20)-P(2)	114.6(2)
C(12)-C(11)-H(11)	105.3	C(21A)-C(20)-H(20)	95.1
P(1)-C(11)-H(11)	105.3	C(21B)-C(20)-H(20)	68.5
C(11)-C(12)-H(12A)	109.5	C(19)-C(20)-H(20)	95.1
C(11)-C(12)-H(12B)	109.5	P(2)-C(20)-H(20)	95.1
H(12A)-C(12)-H(12B)	109.5	C(20)-C(21A)-H(21A)	109.5
C(11)-C(12)-H(12C)	109.5	C(20)-C(21A)-H(21B)	109.5
H(12A)-C(12)-H(12C)	109.5	C(20)-C(21A)-H(21C)	109.5
H(12B)-C(12)-H(12C)	109.5	C(20)-C(21B)-H(21D)	109.5
C(14)-C(13)-H(13A)	109.5	C(20)-C(21B)-H(21E)	109.5
C(14)-C(13)-H(13B)	109.5	H(21D)-C(21B)-H(21E)	109.5
H(13A)-C(13)-H(13B)	109.5	C(20)-C(21B)-H(21F)	109.5
C(14)-C(13)-H(13C)	109.5	H(21D)-C(21B)-H(21F)	109.5
H(13A)-C(13)-H(13C)	109.5	H(21E)-C(21B)-H(21F)	109.5
H(13B)-C(13)-H(13C)	109.5	C(23)-C(22)-H(22A)	109.5
C(13)-C(14)-C(15)	112.3(3)	C(23)-C(22)-H(22B)	109.5
C(13)-C(14)-P(1)	113.3(2)	H(22A)-C(22)-H(22B)	109.5
C(15)-C(14)-P(1)	118.4(2)	C(23)-C(22)-H(22C)	109.5
C(13)-C(14)-H(14)	103.6	H(22A)-C(22)-H(22C)	109.5
C(15)-C(14)-H(14)	103.6	H(22B)-C(22)-H(22C)	109.5
P(1)-C(14)-H(14)	103.6	C(22)-C(23)-C(24)	109.3(3)
C(14)-C(15)-H(15A)	109.5	C(22)-C(23)-P(3)	111.8(2)
C(14)-C(15)-H(15B)	109.5	C(24)-C(23)-P(3)	118.9(3)
H(15A)-C(15)-H(15B)	109.5	C(22)-C(23)-H(23)	105.2
C(14)-C(15)-H(15C)	109.5	C(24)-C(23)-H(23)	105.2
H(15A)-C(15)-H(15C)	109.5	P(3)-C(23)-H(23)	105.2
H(15B)-C(15)-H(15C)	109.5	C(23)-C(24)-H(24A)	109.5
C(17)-C(16)-H(16A)	109.5	C(23)-C(24)-H(24B)	109.5
C(17)-C(16)-H(16B)	109.5	H(24A)-C(24)-H(24B)	109.5
H(16A)-C(16)-H(16B)	109.5	C(23)-C(24)-H(24C)	109.5
C(17)-C(16)-H(16C)	109.5	H(24A)-C(24)-H(24C)	109.5
H(16A)-C(16)-H(16C)	109.5	H(24B)-C(24)-H(24C)	109.5
H(16B)-C(16)-H(16C)	109.5	C(26)-C(25)-H(25A)	109.5
C(16)-C(17)-C(18)	110.0(3)	C(26)-C(25)-H(25B)	109.5
C(16)-C(17)-P(2)	112.2(2)	H(25A)-C(25)-H(25B)	109.5
C(18)-C(17)-P(2)	117.6(2)	C(26)-C(25)-H(25C)	109.5
C(16)-C(17)-H(17)	105.3	H(25A)-C(25)-H(25C)	109.5
C(18)-C(17)-H(17)	105.3	H(25B)-C(25)-H(25C)	109.5
P(2)-C(17)-H(17)	105.3	C(27)-C(26)-C(25)	118.5(3)
C(17)-C(18)-H(18A)	109.5	C(27)-C(26)-P(3)	120.4(3)
C(17)-C(18)-H(18B)	109.5	C(25)-C(26)-P(3)	114.5(3)
H(18A)-C(18)-H(18B)	109.5	C(27)-C(26)-H(26)	98.6
C(17)-C(18)-H(18C)	109.5	C(25)-C(26)-H(26)	98.6
H(18A)-C(18)-H(18C)	109.5	P(3)-C(26)-H(26)	98.6
H(18B)-C(18)-H(18C)	109.5	C(26)-C(27)-H(27A)	109.5
C(20)-C(19)-H(19A)	109.5	C(26)-C(27)-H(27B)	109.5
C(20)-C(19)-H(19B)	109.5	H(27A)-C(27)-H(27B)	109.5
H(19A)-C(19)-H(19B)	109.5	C(26)-C(27)-H(27C)	109.5
C(20)-C(19)-H(19C)	109.5	H(27A)-C(27)-H(27C)	109.5

H(27B)-C(27)-H(27C)	109.5	C(34)-C(33)-H(33B)	111.5
O(2)#2-Na(1)-O(2)	180.0	H(33A)-C(33)-H(33B)	109.3
O(2)#2-Na(1)-O(1)	93.13(9)	C(33)-C(34)-C(35)	103.9(3)
O(2)-Na(1)-O(1)	86.87(9)	C(33)-C(34)-H(34A)	111.0
O(2)#2-Na(1)-O(1)#2	86.87(9)	C(35)-C(34)-H(34A)	111.0
O(2)-Na(1)-O(1)#2	93.13(9)	C(33)-C(34)-H(34B)	111.0
O(1)-Na(1)-O(1)#2	180.000(2)	C(35)-C(34)-H(34B)	111.0
O(2)#2-Na(1)-O(3)	91.13(9)	H(34A)-C(34)-H(34B)	109.0
O(2)-Na(1)-O(3)	88.87(9)	O(2)-C(35)-C(34)	105.8(3)
O(1)-Na(1)-O(3)	87.75(9)	O(2)-C(35)-H(35A)	110.6
O(1)#2-Na(1)-O(3)	92.25(9)	C(34)-C(35)-H(35A)	110.6
O(2)#2-Na(1)-O(3)#2	88.87(9)	O(2)-C(35)-H(35B)	110.6
O(2)-Na(1)-O(3)#2	91.13(9)	C(34)-C(35)-H(35B)	110.6
O(1)-Na(1)-O(3)#2	92.25(9)	H(35A)-C(35)-H(35B)	108.7
O(1)#2-Na(1)-O(3)#2	87.75(9)	C(36)-O(3)-C(39)	107.6(3)
O(3)-Na(1)-O(3)#2	180.000(1)	C(36)-O(3)-Na(1)	124.5(2)
C(28)-O(1)-C(31)	108.4(3)	C(39)-O(3)-Na(1)	127.8(3)
C(28)-O(1)-Na(1)	123.0(3)	O(3)-C(36)-C(37)	106.1(3)
C(31)-O(1)-Na(1)	128.5(2)	O(3)-C(36)-H(36A)	110.5
O(1)-C(28)-C(29)	107.8(4)	C(37)-C(36)-H(36A)	110.5
O(1)-C(28)-H(28A)	110.2	O(3)-C(36)-H(36B)	110.5
C(29)-C(28)-H(28A)	110.2	C(37)-C(36)-H(36B)	110.5
O(1)-C(28)-H(28B)	110.2	H(36A)-C(36)-H(36B)	108.7
C(29)-C(28)-H(28B)	110.2	C(36)-C(37)-C(38)	103.0(3)
H(28A)-C(28)-H(28B)	108.5	C(36)-C(37)-H(37A)	111.2
C(30)-C(29)-C(28)	105.8(4)	C(38)-C(37)-H(37A)	111.2
C(30)-C(29)-H(29A)	110.6	C(36)-C(37)-H(37B)	111.2
C(28)-C(29)-H(29A)	110.6	C(38)-C(37)-H(37B)	111.2
C(30)-C(29)-H(29B)	110.6	H(37A)-C(37)-H(37B)	109.1
C(28)-C(29)-H(29B)	110.6	C(39)-C(38)-C(37)	106.2(4)
H(29A)-C(29)-H(29B)	108.7	C(39)-C(38)-H(38A)	110.5
C(29)-C(30)-C(31)	109.7(5)	C(37)-C(38)-H(38A)	110.5
C(29)-C(30)-H(30A)	109.7	C(39)-C(38)-H(38B)	110.5
C(31)-C(30)-H(30A)	109.7	C(37)-C(38)-H(38B)	110.5
C(29)-C(30)-H(30B)	109.7	H(38A)-C(38)-H(38B)	108.7
C(31)-C(30)-H(30B)	109.7	C(38)-C(39)-O(3)	110.7(4)
H(30A)-C(30)-H(30B)	108.2	C(38)-C(39)-H(39A)	109.5
O(1)-C(31)-C(30)	105.5(4)	O(3)-C(39)-H(39A)	109.5
O(1)-C(31)-H(31A)	110.6	C(38)-C(39)-H(39B)	109.5
C(30)-C(31)-H(31A)	110.7	O(3)-C(39)-H(39B)	109.5
O(1)-C(31)-H(31B)	110.7	H(39A)-C(39)-H(39B)	108.1
C(30)-C(31)-H(31B)	110.7	C(43)-O(4)-C(40)	101.0(6)
H(31A)-C(31)-H(31B)	108.8	O(4)-C(40)-C(41)	99.2(5)
C(32)-O(2)-C(35)	109.4(3)	O(4)-C(40)-H(40A)	111.8
C(32)-O(2)-Na(1)	125.8(2)	C(41)-C(40)-H(40A)	111.8
C(35)-O(2)-Na(1)	124.8(2)	O(4)-C(40)-H(40B)	112.0
O(2)-C(32)-C(33)	107.6(3)	C(41)-C(40)-H(40B)	112.2
O(2)-C(32)-H(32A)	110.2	H(40A)-C(40)-H(40B)	109.6
C(33)-C(32)-H(32A)	110.2	C(42)-C(41)-C(40)	89.4(8)
O(2)-C(32)-H(32B)	110.2	C(42)-C(41)-H(41A)	114.1
C(33)-C(32)-H(32B)	110.2	C(40)-C(41)-H(41A)	113.7
H(32A)-C(32)-H(32B)	108.5	C(42)-C(41)-H(41B)	113.7
C(32)-C(33)-C(34)	101.4(3)	C(40)-C(41)-H(41B)	113.6
C(32)-C(33)-H(33A)	111.5	H(41A)-C(41)-H(41B)	111.0
C(34)-C(33)-H(33A)	111.5	C(41)-C(42)-C(43)	104.0(8)
C(32)-C(33)-H(33B)	111.5	C(41)-C(42)-H(42A)	111.3

C(43)-C(42)-H(42A)	111.4	C(42)-C(43)-H(43A)	110.5
C(41)-C(42)-H(42B)	110.5	O(4)-C(43)-H(43A)	110.8
C(43)-C(42)-H(42B)	110.7	C(42)-C(43)-H(43B)	111.2
H(42A)-C(42)-H(42B)	108.9	O(4)-C(43)-H(43B)	111.0
C(42)-C(43)-O(4)	104.5(7)	H(43A)-C(43)-H(43B)	108.9

Symmetry transformations used to generate equivalent atoms:
#1 -x+2,-y+1,-z+1 #2 -x+2,-y+2,-z+2

Table 9. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for [{$(PhBP^{iPr}_3)Co\}_2(\mu^2\text{-}N_2)$][$Na(THF)_6$]·THF. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^2U^{11} + ...\ + 2\ h\ k\ a\ b\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Co(1)	21(1)	22(1)	27(1)	-12(1)	3(1)	-3(1)
P(1)	22(1)	24(1)	23(1)	-11(1)	2(1)	-6(1)
P(2)	24(1)	27(1)	29(1)	-13(1)	8(1)	-9(1)
P(3)	24(1)	21(1)	28(1)	-10(1)	1(1)	-3(1)
N(1)	21(2)	23(2)	23(2)	-7(1)	3(1)	-7(1)
B(1)	25(2)	24(2)	22(2)	-11(2)	2(2)	-5(2)
C(1)	22(2)	26(2)	25(2)	-10(1)	1(1)	-7(1)
C(2)	33(2)	28(2)	27(2)	-7(2)	-1(2)	0(2)
C(3)	44(2)	45(2)	24(2)	-14(2)	2(2)	1(2)
C(4)	38(2)	33(2)	35(2)	-19(2)	-1(2)	1(2)
C(5)	32(2)	26(2)	36(2)	-11(2)	1(2)	-1(2)
C(6)	33(2)	28(2)	26(2)	-7(2)	5(2)	-5(2)
C(7)	21(2)	21(2)	24(2)	-8(1)	4(1)	-3(1)
C(8)	26(2)	22(2)	25(2)	-10(1)	3(1)	-6(1)
C(9)	23(2)	27(2)	22(2)	-11(1)	3(1)	-6(1)
C(10)	37(2)	31(2)	55(3)	-10(2)	0(2)	-14(2)
C(11)	31(2)	29(2)	32(2)	-8(2)	-2(2)	-12(2)
C(12)	43(2)	41(2)	93(4)	-12(2)	20(2)	-18(2)
C(13)	60(3)	41(2)	28(2)	-11(2)	-8(2)	-11(2)
C(14)	28(2)	38(2)	28(2)	-9(2)	-2(2)	-6(2)
C(15)	32(2)	71(3)	35(2)	-15(2)	-1(2)	-13(2)
C(16)	37(2)	57(2)	29(2)	-9(2)	7(2)	-18(2)
C(17)	40(2)	34(2)	29(2)	-6(2)	8(2)	-18(2)
C(18)	97(4)	40(2)	47(3)	2(2)	-2(2)	-28(2)
C(19)	30(2)	66(3)	88(3)	-51(3)	18(2)	-18(2)
C(20)	34(2)	85(3)	108(4)	-69(3)	33(2)	-35(2)
C(21A)	53(8)	49(11)	85(13)	-42(8)	27(8)	-33(9)
C(21B)	38(6)	27(8)	86(13)	-17(6)	-2(7)	-19(7)
C(22)	26(2)	54(2)	55(3)	-21(2)	-8(2)	-3(2)
C(23)	26(2)	31(2)	43(2)	-11(2)	-5(2)	-4(2)
C(24)	38(3)	139(5)	71(3)	-64(3)	-9(2)	-14(3)
C(25)	69(3)	25(2)	53(3)	-10(2)	2(2)	-10(2)
C(26)	65(3)	30(2)	34(2)	-7(2)	11(2)	-13(2)
C(27)	144(5)	40(3)	43(3)	-8(2)	16(3)	-25(3)
Na(1)	36(1)	37(1)	25(1)	-3(1)	-1(1)	-1(1)
O(1)	48(2)	50(2)	52(2)	-20(1)	18(1)	-13(1)
C(28)	92(4)	125(5)	138(5)	-77(4)	82(4)	-66(4)
C(29)	86(4)	127(5)	92(4)	-58(4)	55(3)	-55(4)
C(30)	226(8)	71(4)	151(6)	-30(4)	145(6)	-19(5)
C(31)	92(4)	50(3)	72(3)	-20(2)	38(3)	-16(3)
O(2)	53(2)	51(2)	36(2)	6(1)	-10(1)	-18(1)
C(32)	57(3)	58(3)	57(3)	12(2)	-9(2)	-10(2)
C(33)	46(2)	44(2)	58(3)	-1(2)	2(2)	-7(2)
C(34)	67(3)	54(3)	63(3)	-25(2)	9(2)	-13(2)
C(35)	68(3)	82(3)	39(3)	-2(2)	0(2)	-27(3)
O(3)	54(2)	59(2)	31(2)	-5(1)	-15(1)	3(1)
C(36)	57(3)	64(3)	51(3)	-3(2)	-17(2)	-2(2)
C(37)	83(3)	46(3)	67(3)	8(2)	-42(3)	-23(2)
C(38)	80(4)	100(4)	65(3)	-22(3)	-33(3)	-2(3)
C(39)	130(5)	95(4)	37(3)	-19(3)	-28(3)	11(3)
O(4)	109(4)	146(4)	209(6)	7(4)	-20(4)	-26(3)

C(40)	121(7)	402(15)	119(6)	168(8)	-44(5)	-123(8)
C(41)	83(6)	186(9)	218(9)	-45(7)	-46(6)	11(5)
C(42)	251(13)	115(7)	520(20)	47(10)	-245(14)	-106(8)
C(43)	103(6)	261(11)	414(16)	230(11)	-44(8)	-115(7)

Table 10. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for [{$(PhBP^{iPr}_3)Co\}_2(\mu^2\text{-}N_2)$][$Na(THF)_6$]·THF.

	x	y	z	U(eq)
H(2)	6184	8072	8791	37
H(3)	5098	9324	9584	48
H(4)	4276	10914	8834	44
H(5)	4496	11202	7272	40
H(6)	5587	9959	6490	36
H(7A)	5664	8503	5800	27
H(7B)	5150	7863	6594	27
H(8A)	8584	8304	6876	29
H(8B)	7619	9099	6213	29
H(9A)	6782	6701	7869	29
H(9B)	7792	7099	8155	29
H(10A)	6070	4481	6404	60
H(10B)	7151	4798	5866	60
H(10C)	5905	5004	5379	60
H(11)	6029	5849	6904	36
H(12A)	4056	6217	5766	89
H(12B)	4027	6940	6457	89
H(12C)	4119	5832	6826	89
H(13A)	5755	6066	4057	65
H(13B)	7031	6260	3903	65
H(13C)	5922	6897	3271	65
H(14)	6087	7828	4255	39
H(15A)	4169	8252	3940	70
H(15B)	4139	8223	5008	70
H(15C)	3953	7346	4602	70
H(16A)	7751	8630	3146	61
H(16B)	8231	7554	3704	61
H(16C)	9130	8183	3454	61
H(17)	7043	8693	4607	40
H(18A)	8750	9692	4122	92
H(18B)	7809	9916	4955	92
H(18C)	7321	10099	3957	92
H(19A)	11935	7248	4816	86
H(19B)	10842	7301	4216	86
H(19C)	11196	6496	5096	86
H(20)	10089	8422	4884	80
H(21A)	10845	7888	6546	84
H(21B)	10112	8936	6017	84
H(21C)	11475	8382	5705	84
H(21D)	11430	8459	5782	71
H(21E)	10062	8910	6143	71
H(21F)	10427	9207	5113	71
H(22A)	11903	5524	7217	68
H(22B)	11080	5425	6466	68
H(22C)	11267	4697	7401	68
H(23)	10099	6735	7265	40
H(24A)	10396	5283	8809	120
H(24B)	9472	6367	8812	120
H(24C)	10895	6190	8644	120
H(25A)	10010	3805	7340	75

H(25B)	8975	4154	6608	75
H(25C)	8852	3440	7513	75
H(26)	7639	4939	7536	53
H(27A)	7954	4163	8872	117
H(27B)	7760	5290	8889	117
H(27C)	9093	4506	8978	117
H(28A)	13000	9173	8982	130
H(28B)	12017	9832	8251	130
H(29A)	12525	8713	7372	114
H(29B)	13745	8251	7939	114
H(30A)	13188	7123	8618	197
H(30B)	12182	7427	7865	197
H(31A)	10743	7931	8840	89
H(31B)	11737	7503	9612	89
H(32A)	9450	8301	11654	74
H(32B)	10117	7526	10987	74
H(33A)	7673	8001	11652	62
H(33B)	8600	6937	11513	62
H(34A)	7024	7863	10305	74
H(34B)	8368	7238	9990	74
H(35A)	8377	8691	9230	76
H(35B)	7380	9289	9906	76
H(36A)	7731	11933	8941	74
H(36B)	7020	11161	8969	74
H(37A)	7711	12279	7440	78
H(37B)	6544	11911	7570	78
H(38A)	7595	10459	7169	102
H(38B)	8489	11024	6667	102
H(39A)	9827	10319	7599	116
H(39B)	9070	9587	7933	116
H(40A)	5277	4196	8083	273
H(40B)	5598	4960	8594	273
H(41A)	3809	4989	9651	206
H(41B)	3308	5280	8628	206
H(42A)	3990	3718	8473	336
H(42B)	3048	3869	9322	336
H(43A)	4979	2638	9597	334
H(43B)	4535	3418	10289	334

Figure 3. Fully labeled drawing of of [PhBP$^{iPr}_3$]Fe≡NAd (**9**)

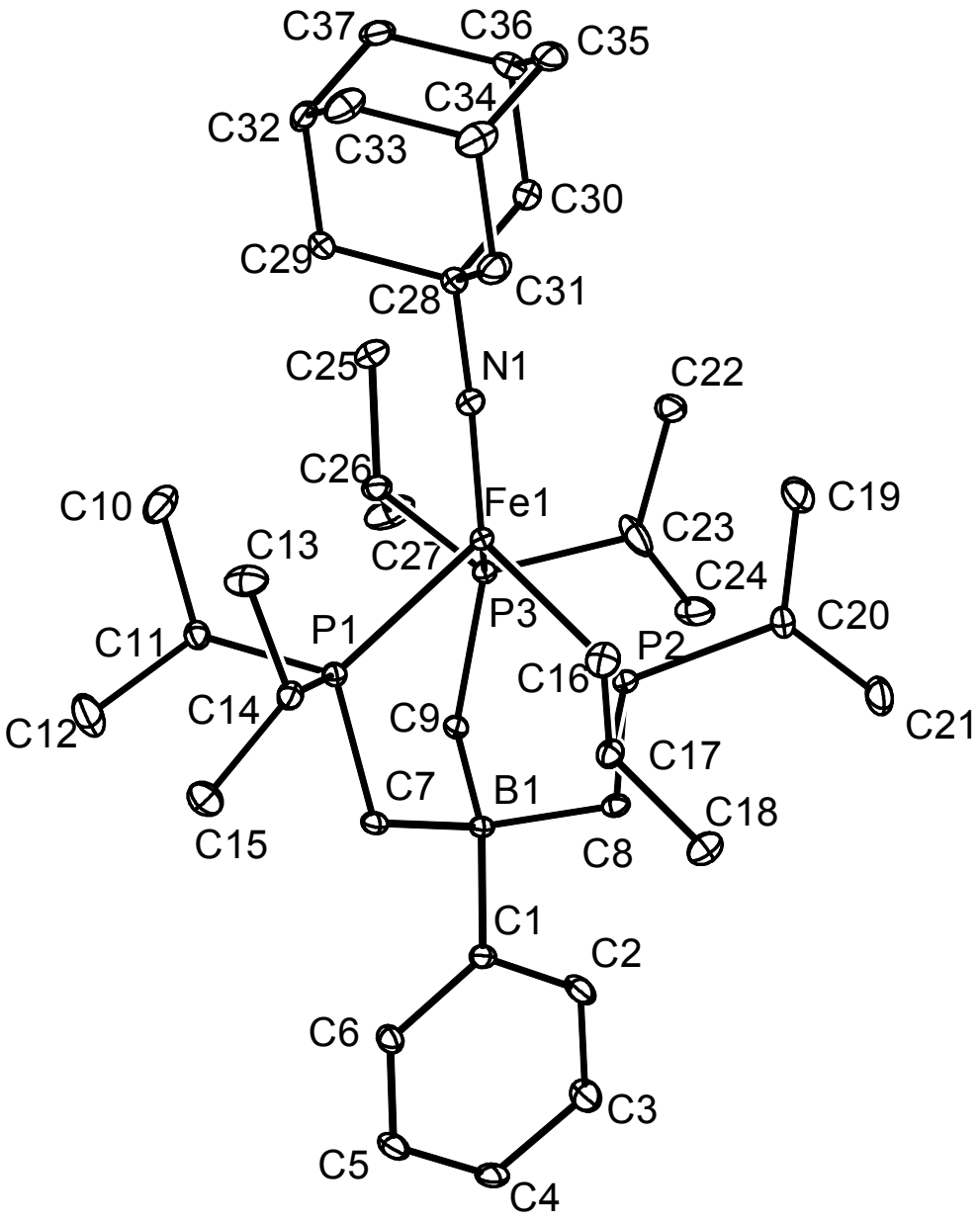

Table 11. Crystal data and structure refinement for [$PhBP^{iPr}_3$]Fe≡NAd.

Identification code	tab40	
Empirical formula	$C_{37}H_{68}BFeNP_3$	
Formula weight	686.49	
Temperature	96(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/n	
Unit cell dimensions	a = 12.5232(9) Å	α= 90°.
	b = 16.2311(11) Å	β= 101.9480(10)°.
	c = 18.8942(13) Å	γ = 90°.
Volume	3757.3(5) $Å^3$	
Z	4	
Density (calculated)	1.214 Mg/m^3	
Absorption coefficient	0.555 mm^{-1}	
F(000)	1492	
Crystal size	0.11 x 0.25 x 0.31 mm^3	
Theta range for data collection	1.67 to 28.49°.	
Index ranges	-15<=h<=16, -21<=k<=21, -23<=l<=25	
Reflections collected	55648	
Independent reflections	8921 [R(int) = 0.0704]	
Completeness to theta = 28.49°	93.6 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	8921 / 0 / 400	
Goodness-of-fit on F^2	1.805	
Final R indices [I>2sigma(I)]	R1 = 0.0495, wR2 = 0.0964	
R indices (all data)	R1 = 0.0795, wR2 = 0.1018	
Largest diff. peak and hole	1.140 and -1.002 $e.Å^{-3}$	

Special Refinement Details

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Table 12. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [$PhBP^{iPr}_3$]Fe≡NAd. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Fe(1)	915(1)	631(1)	2489(1)	21(1)
P(1)	1239(1)	998(1)	3668(1)	19(1)
P(2)	-432(1)	-263(1)	2656(1)	18(1)
P(3)	-285(1)	1679(1)	2184(1)	22(1)
N(1)	1911(2)	501(1)	2058(1)	26(1)
B(1)	-1161(2)	1131(2)	3439(1)	19(1)
C(1)	-2148(2)	1400(1)	3841(1)	20(1)
C(2)	-3232(2)	1353(2)	3473(1)	37(1)
C(3)	-4105(2)	1599(2)	3767(2)	40(1)
C(4)	-3935(2)	1906(2)	4458(1)	28(1)
C(5)	-2880(2)	1975(2)	4840(1)	27(1)
C(6)	-2015(2)	1727(2)	4533(1)	24(1)
C(7)	4(2)	1020(2)	4038(1)	23(1)
C(8)	-1518(2)	256(1)	2992(1)	18(1)
C(9)	-1072(2)	1895(1)	2871(1)	20(1)
C(10)	2712(2)	2235(2)	3414(2)	41(1)
C(11)	1788(2)	2062(1)	3813(1)	21(1)
C(12)	2109(3)	2362(2)	4588(2)	46(1)
C(13)	3327(2)	242(2)	4066(2)	38(1)
C(14)	2210(2)	298(2)	4270(1)	22(1)
C(15)	2335(2)	427(2)	5081(1)	33(1)
C(16)	1073(2)	-1505(2)	3190(1)	30(1)
C(17)	66(2)	-1065(1)	3348(1)	22(1)
C(18)	-789(2)	-1694(2)	3475(1)	30(1)
C(19)	-409(2)	-953(2)	1273(2)	48(1)
C(20)	-1030(2)	-897(2)	1839(2)	50(1)
C(21)	-2190(2)	-1091(2)	1699(2)	44(1)
C(22)	-794(2)	1278(2)	704(1)	28(1)
C(23)	-1282(2)	1402(3)	1348(2)	67(1)
C(24)	-2428(2)	1562(2)	1273(1)	38(1)
C(25)	1064(2)	2706(2)	1503(2)	37(1)
C(26)	265(2)	2710(2)	2008(1)	23(1)
C(27)	-611(2)	3365(2)	1773(2)	45(1)
C(28)	2796(2)	448(1)	1682(1)	23(1)
C(29)	3691(2)	1070(2)	1995(1)	25(1)
C(30)	2387(2)	635(2)	874(1)	33(1)
C(31)	3290(2)	-420(2)	1768(2)	34(1)
C(32)	4639(2)	1012(2)	1598(1)	26(1)
C(33)	5115(2)	148(2)	1689(2)	38(1)
C(34)	4237(2)	-479(2)	1373(2)	44(1)
C(35)	3820(3)	-297(2)	579(2)	52(1)
C(36)	3335(2)	572(2)	478(1)	39(1)
C(37)	4217(2)	1199(2)	802(1)	31(1)

Table 13. Bond lengths [Å] and angles [°] for [$PhBP^{iPr}_3$]Fe≡NAd.

Fe(1)-N(1)	1.638(2)	C(16)-H(16A)	0.9800
Fe(1)-P(1)	2.2603(7)	C(16)-H(16B)	0.9800
Fe(1)-P(3)	2.2634(8)	C(16)-H(16C)	0.9800
Fe(1)-P(2)	2.2969(7)	C(17)-C(18)	1.534(3)
P(1)-C(7)	1.826(2)	C(17)-H(17)	1.0000
P(1)-C(11)	1.858(2)	C(18)-H(18A)	0.9800
P(1)-C(14)	1.869(2)	C(18)-H(18B)	0.9800
P(2)-C(8)	1.823(2)	C(18)-H(18C)	0.9800
P(2)-C(17)	1.859(2)	C(19)-C(20)	1.450(4)
P(2)-C(20)	1.876(3)	C(19)-H(19A)	0.9800
P(3)-C(9)	1.820(2)	C(19)-H(19B)	0.9800
P(3)-C(23)	1.855(3)	C(19)-H(19C)	0.9800
P(3)-C(26)	1.865(2)	C(20)-C(21)	1.456(3)
N(1)-C(28)	1.438(3)	C(20)-H(20)	1.0000
B(1)-C(1)	1.639(3)	C(21)-H(21A)	0.9800
B(1)-C(9)	1.659(3)	C(21)-H(21B)	0.9800
B(1)-C(7)	1.660(3)	C(21)-H(21C)	0.9800
B(1)-C(8)	1.665(3)	C(22)-C(23)	1.485(3)
C(1)-C(6)	1.388(3)	C(22)-H(22A)	0.9800
C(1)-C(2)	1.393(3)	C(22)-H(22B)	0.9800
C(2)-C(3)	1.383(3)	C(22)-H(22C)	0.9800
C(2)-H(2)	0.9500	C(23)-C(24)	1.435(3)
C(3)-C(4)	1.371(3)	C(23)-H(23)	1.0000
C(3)-H(3)	0.9500	C(24)-H(24A)	0.9800
C(4)-C(5)	1.373(3)	C(24)-H(24B)	0.9800
C(4)-H(4)	0.9500	C(24)-H(24C)	0.9800
C(5)-C(6)	1.391(3)	C(25)-C(26)	1.519(3)
C(5)-H(5)	0.9500	C(25)-H(25A)	0.9800
C(6)-H(6)	0.9500	C(25)-H(25B)	0.9800
C(7)-H(7A)	0.9900	C(25)-H(25C)	0.9800
C(7)-H(7B)	0.9900	C(26)-C(27)	1.526(3)
C(8)-H(8A)	0.9900	C(26)-H(26)	1.0000
C(8)-H(8B)	0.9900	C(27)-H(27A)	0.9800
C(9)-H(9A)	0.9900	C(27)-H(27B)	0.9800
C(9)-H(9B)	0.9900	C(27)-H(27C)	0.9800
C(10)-C(11)	1.531(3)	C(28)-C(29)	1.533(3)
C(10)-H(10A)	0.9800	C(28)-C(31)	1.534(3)
C(10)-H(10B)	0.9800	C(28)-C(30)	1.537(3)
C(10)-H(10C)	0.9800	C(29)-C(32)	1.533(3)
C(11)-C(12)	1.517(3)	C(29)-H(29A)	0.9900
C(11)-H(11)	1.0000	C(29)-H(29B)	0.9900
C(12)-H(12A)	0.9800	C(30)-C(36)	1.533(3)
C(12)-H(12B)	0.9800	C(30)-H(30A)	0.9900
C(12)-H(12C)	0.9800	C(30)-H(30B)	0.9900
C(13)-C(14)	1.528(3)	C(31)-C(34)	1.529(4)
C(13)-H(13A)	0.9800	C(31)-H(31A)	0.9900
C(13)-H(13B)	0.9800	C(31)-H(31B)	0.9900
C(13)-H(13C)	0.9800	C(32)-C(37)	1.518(4)
C(14)-C(15)	1.522(3)	C(32)-C(33)	1.519(3)
C(14)-H(14)	1.0000	C(32)-H(32)	1.0000
C(15)-H(15A)	0.9800	C(33)-C(34)	1.527(4)
C(15)-H(15B)	0.9800	C(33)-H(33A)	0.9900
C(15)-H(15C)	0.9800	C(33)-H(33B)	0.9900
C(16)-C(17)	1.530(3)	C(34)-C(35)	1.512(4)

C(34)-H(34)	1.0000	C(5)-C(6)-H(6)	118.3
C(35)-C(36)	1.532(4)	B(1)-C(7)-P(1)	115.79(15)
C(35)-H(35A)	0.9900	B(1)-C(7)-H(7A)	108.3
C(35)-H(35B)	0.9900	P(1)-C(7)-H(7A)	108.3
C(36)-C(37)	1.532(4)	B(1)-C(7)-H(7B)	108.3
C(36)-H(36)	1.0000	P(1)-C(7)-H(7B)	108.3
C(37)-H(37A)	0.9900	H(7A)-C(7)-H(7B)	107.4
C(37)-H(37B)	0.9900	B(1)-C(8)-P(2)	115.43(15)
N(1)-Fe(1)-P(1)	121.62(7)	B(1)-C(8)-H(8A)	108.4
N(1)-Fe(1)-P(3)	120.27(7)	P(2)-C(8)-H(8A)	108.4
P(1)-Fe(1)-P(3)	91.56(3)	B(1)-C(8)-H(8B)	108.4
N(1)-Fe(1)-P(2)	130.03(7)	P(2)-C(8)-H(8B)	108.4
P(1)-Fe(1)-P(2)	91.05(2)	H(8A)-C(8)-H(8B)	107.5
P(3)-Fe(1)-P(2)	92.39(3)	B(1)-C(9)-P(3)	115.82(16)
C(7)-P(1)-C(11)	104.04(11)	B(1)-C(9)-H(9A)	108.3
C(7)-P(1)-C(14)	105.94(11)	P(3)-C(9)-H(9A)	108.3
C(11)-P(1)-C(14)	107.56(11)	B(1)-C(9)-H(9B)	108.3
C(7)-P(1)-Fe(1)	112.91(8)	P(3)-C(9)-H(9B)	108.3
C(11)-P(1)-Fe(1)	112.06(8)	H(9A)-C(9)-H(9B)	107.4
C(14)-P(1)-Fe(1)	113.66(8)	C(11)-C(10)-H(10A)	109.5
C(8)-P(2)-C(17)	104.15(10)	C(11)-C(10)-H(10B)	109.5
C(8)-P(2)-C(20)	109.93(12)	H(10A)-C(10)-H(10B)	109.5
C(17)-P(2)-C(20)	102.31(14)	C(11)-C(10)-H(10C)	109.5
C(8)-P(2)-Fe(1)	112.22(8)	H(10A)-C(10)-H(10C)	109.5
C(17)-P(2)-Fe(1)	112.95(8)	H(10B)-C(10)-H(10C)	109.5
C(20)-P(2)-Fe(1)	114.40(9)	C(12)-C(11)-C(10)	109.9(2)
C(9)-P(3)-C(23)	106.61(12)	C(12)-C(11)-P(1)	117.14(18)
C(9)-P(3)-C(26)	103.14(11)	C(10)-C(11)-P(1)	113.06(16)
C(23)-P(3)-C(26)	105.91(16)	C(12)-C(11)-H(11)	105.2
C(9)-P(3)-Fe(1)	113.12(8)	C(10)-C(11)-H(11)	105.2
C(23)-P(3)-Fe(1)	109.24(12)	P(1)-C(11)-H(11)	105.2
C(26)-P(3)-Fe(1)	118.01(8)	C(11)-C(12)-H(12A)	109.5
C(28)-N(1)-Fe(1)	175.98(17)	C(11)-C(12)-H(12B)	109.5
C(1)-B(1)-C(9)	105.00(19)	H(12A)-C(12)-H(12B)	109.5
C(1)-B(1)-C(7)	110.66(18)	C(11)-C(12)-H(12C)	109.5
C(9)-B(1)-C(7)	110.79(19)	H(12A)-C(12)-H(12C)	109.5
C(1)-B(1)-C(8)	108.21(18)	H(12B)-C(12)-H(12C)	109.5
C(9)-B(1)-C(8)	110.95(18)	C(14)-C(13)-H(13A)	109.5
C(7)-B(1)-C(8)	111.03(19)	C(14)-C(13)-H(13B)	109.5
C(6)-C(1)-C(2)	113.9(2)	H(13A)-C(13)-H(13B)	109.5
C(6)-C(1)-B(1)	125.7(2)	C(14)-C(13)-H(13C)	109.5
C(2)-C(1)-B(1)	120.4(2)	H(13A)-C(13)-H(13C)	109.5
C(3)-C(2)-C(1)	123.6(2)	H(13B)-C(13)-H(13C)	109.5
C(3)-C(2)-H(2)	118.2	C(15)-C(14)-C(13)	110.7(2)
C(1)-C(2)-H(2)	118.2	C(15)-C(14)-P(1)	116.57(17)
C(4)-C(3)-C(2)	120.4(2)	C(13)-C(14)-P(1)	113.37(17)
C(4)-C(3)-H(3)	119.8	C(15)-C(14)-H(14)	105.0
C(2)-C(3)-H(3)	119.8	C(13)-C(14)-H(14)	105.0
C(3)-C(4)-C(5)	118.3(2)	P(1)-C(14)-H(14)	105.0
C(3)-C(4)-H(4)	120.9	C(14)-C(15)-H(15A)	109.5
C(5)-C(4)-H(4)	120.9	C(14)-C(15)-H(15B)	109.5
C(4)-C(5)-C(6)	120.3(2)	H(15A)-C(15)-H(15B)	109.5
C(4)-C(5)-H(5)	119.8	C(14)-C(15)-H(15C)	109.5
C(6)-C(5)-H(5)	119.8	H(15A)-C(15)-H(15C)	109.5
C(1)-C(6)-C(5)	123.5(2)	H(15B)-C(15)-H(15C)	109.5
C(1)-C(6)-H(6)	118.3	C(17)-C(16)-H(16A)	109.5

C(17)-C(16)-H(16B)	109.5	C(26)-C(25)-H(25C)	109.5
H(16A)-C(16)-H(16B)	109.5	H(25A)-C(25)-H(25C)	109.5
C(17)-C(16)-H(16C)	109.5	H(25B)-C(25)-H(25C)	109.5
H(16A)-C(16)-H(16C)	109.5	C(25)-C(26)-C(27)	110.4(2)
H(16B)-C(16)-H(16C)	109.5	C(25)-C(26)-P(3)	115.05(18)
C(16)-C(17)-C(18)	110.5(2)	C(27)-C(26)-P(3)	114.06(17)
C(16)-C(17)-P(2)	111.08(17)	C(25)-C(26)-H(26)	105.5
C(18)-C(17)-P(2)	115.63(16)	C(27)-C(26)-H(26)	105.5
C(16)-C(17)-H(17)	106.3	P(3)-C(26)-H(26)	105.5
C(18)-C(17)-H(17)	106.3	C(26)-C(27)-H(27A)	109.5
P(2)-C(17)-H(17)	106.3	C(26)-C(27)-H(27B)	109.5
C(17)-C(18)-H(18A)	109.5	H(27A)-C(27)-H(27B)	109.5
C(17)-C(18)-H(18B)	109.5	C(26)-C(27)-H(27C)	109.5
H(18A)-C(18)-H(18B)	109.5	H(27A)-C(27)-H(27C)	109.5
C(17)-C(18)-H(18C)	109.5	H(27B)-C(27)-H(27C)	109.5
H(18A)-C(18)-H(18C)	109.5	N(1)-C(28)-C(29)	110.27(18)
H(18B)-C(18)-H(18C)	109.5	N(1)-C(28)-C(31)	109.74(19)
C(20)-C(19)-H(19A)	109.5	C(29)-C(28)-C(31)	108.5(2)
C(20)-C(19)-H(19B)	109.5	N(1)-C(28)-C(30)	110.2(2)
H(19A)-C(19)-H(19B)	109.5	C(29)-C(28)-C(30)	108.67(19)
C(20)-C(19)-H(19C)	109.5	C(31)-C(28)-C(30)	109.4(2)
H(19A)-C(19)-H(19C)	109.5	C(32)-C(29)-C(28)	110.44(19)
H(19B)-C(19)-H(19C)	109.5	C(32)-C(29)-H(29A)	109.6
C(19)-C(20)-C(21)	121.3(2)	C(28)-C(29)-H(29A)	109.6
C(19)-C(20)-P(2)	117.20(19)	C(32)-C(29)-H(29B)	109.6
C(21)-C(20)-P(2)	118.8(2)	C(28)-C(29)-H(29B)	109.6
C(19)-C(20)-H(20)	95.5	H(29A)-C(29)-H(29B)	108.1
C(21)-C(20)-H(20)	95.5	C(36)-C(30)-C(28)	109.8(2)
P(2)-C(20)-H(20)	95.5	C(36)-C(30)-H(30A)	109.7
C(20)-C(21)-H(21A)	109.5	C(28)-C(30)-H(30A)	109.7
C(20)-C(21)-H(21B)	109.5	C(36)-C(30)-H(30B)	109.7
H(21A)-C(21)-H(21B)	109.5	C(28)-C(30)-H(30B)	109.7
C(20)-C(21)-H(21C)	109.5	H(30A)-C(30)-H(30B)	108.2
H(21A)-C(21)-H(21C)	109.5	C(34)-C(31)-C(28)	110.0(2)
H(21B)-C(21)-H(21C)	109.5	C(34)-C(31)-H(31A)	109.7
C(23)-C(22)-H(22A)	109.5	C(28)-C(31)-H(31A)	109.7
C(23)-C(22)-H(22B)	109.5	C(34)-C(31)-H(31B)	109.7
H(22A)-C(22)-H(22B)	109.5	C(28)-C(31)-H(31B)	109.7
C(23)-C(22)-H(22C)	109.5	H(31A)-C(31)-H(31B)	108.2
H(22A)-C(22)-H(22C)	109.5	C(37)-C(32)-C(33)	110.1(2)
H(22B)-C(22)-H(22C)	109.5	C(37)-C(32)-C(29)	109.0(2)
C(24)-C(23)-C(22)	121.1(2)	C(33)-C(32)-C(29)	109.1(2)
C(24)-C(23)-P(3)	121.6(2)	C(37)-C(32)-H(32)	109.5
C(22)-C(23)-P(3)	114.31(19)	C(33)-C(32)-H(32)	109.5
C(24)-C(23)-H(23)	95.8	C(29)-C(32)-H(32)	109.5
C(22)-C(23)-H(23)	95.8	C(32)-C(33)-C(34)	109.8(2)
P(3)-C(23)-H(23)	95.8	C(32)-C(33)-H(33A)	109.7
C(23)-C(24)-H(24A)	109.5	C(34)-C(33)-H(33A)	109.7
C(23)-C(24)-H(24B)	109.5	C(32)-C(33)-H(33B)	109.7
H(24A)-C(24)-H(24B)	109.5	C(34)-C(33)-H(33B)	109.7
C(23)-C(24)-H(24C)	109.5	H(33A)-C(33)-H(33B)	108.2
H(24A)-C(24)-H(24C)	109.5	C(35)-C(34)-C(33)	109.3(2)
H(24B)-C(24)-H(24C)	109.5	C(35)-C(34)-C(31)	109.2(2)
C(26)-C(25)-H(25A)	109.5	C(33)-C(34)-C(31)	109.6(2)
C(26)-C(25)-H(25B)	109.5	C(35)-C(34)-H(34)	109.6
H(25A)-C(25)-H(25B)	109.5	C(33)-C(34)-H(34)	109.6

C(31)-C(34)-H(34)	109.6	C(35)-C(36)-H(36)	109.8
C(34)-C(35)-C(36)	110.6(2)	C(37)-C(36)-H(36)	109.8
C(34)-C(35)-H(35A)	109.5	C(30)-C(36)-H(36)	109.8
C(36)-C(35)-H(35A)	109.5	C(32)-C(37)-C(36)	109.8(2)
C(34)-C(35)-H(35B)	109.5	C(32)-C(37)-H(37A)	109.7
C(36)-C(35)-H(35B)	109.5	C(36)-C(37)-H(37A)	109.7
H(35A)-C(35)-H(35B)	108.1	C(32)-C(37)-H(37B)	109.7
C(35)-C(36)-C(37)	109.1(2)	C(36)-C(37)-H(37B)	109.7
C(35)-C(36)-C(30)	109.1(2)	H(37A)-C(37)-H(37B)	108.2
C(37)-C(36)-C(30)	109.1(2)		

Table 14. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for [$PhBP^{iPr}_3$]Fe≡NAd. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^2U^{11} + ...\ + 2\ h\ k\ a\ b\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Fe(1)	21(1)	27(1)	17(1)	-4(1)	8(1)	-9(1)
P(1)	15(1)	24(1)	17(1)	-4(1)	5(1)	-2(1)
P(2)	16(1)	21(1)	19(1)	-3(1)	7(1)	-3(1)
P(3)	16(1)	31(1)	21(1)	-9(1)	8(1)	-6(1)
N(1)	27(1)	27(1)	25(1)	-3(1)	10(1)	-8(1)
B(1)	16(1)	25(2)	16(1)	-4(1)	7(1)	-2(1)
C(1)	19(1)	25(1)	17(1)	1(1)	6(1)	-1(1)
C(2)	20(1)	69(2)	21(1)	-19(1)	5(1)	0(1)
C(3)	14(1)	74(2)	30(2)	-18(2)	2(1)	-2(1)
C(4)	21(1)	40(2)	26(1)	-5(1)	13(1)	3(1)
C(5)	26(1)	37(2)	18(1)	-6(1)	7(1)	4(1)
C(6)	19(1)	34(2)	20(1)	-6(1)	2(1)	0(1)
C(7)	17(1)	35(1)	18(1)	-6(1)	7(1)	-3(1)
C(8)	13(1)	24(1)	17(1)	4(1)	4(1)	2(1)
C(9)	18(1)	25(1)	19(1)	-8(1)	8(1)	-4(1)
C(10)	27(2)	32(2)	69(2)	-16(2)	21(2)	-8(1)
C(11)	20(1)	21(1)	22(1)	-5(1)	3(1)	0(1)
C(12)	74(2)	33(2)	28(2)	-6(1)	3(2)	-12(2)
C(13)	24(1)	55(2)	37(2)	11(1)	10(1)	12(1)
C(14)	19(1)	23(1)	23(1)	-1(1)	3(1)	-3(1)
C(15)	39(2)	35(2)	24(1)	4(1)	3(1)	4(1)
C(16)	23(1)	28(2)	38(2)	-1(1)	5(1)	8(1)
C(17)	20(1)	19(1)	27(1)	-2(1)	5(1)	-1(1)
C(18)	27(1)	22(1)	41(2)	7(1)	9(1)	2(1)
C(19)	38(2)	67(2)	43(2)	-31(2)	16(1)	-9(2)
C(20)	42(2)	77(2)	36(2)	-33(2)	18(1)	-35(2)
C(21)	26(2)	65(2)	40(2)	-27(2)	3(1)	-9(1)
C(22)	23(1)	42(2)	19(1)	2(1)	3(1)	-2(1)
C(23)	17(2)	140(3)	42(2)	-59(2)	3(1)	-4(2)
C(24)	22(1)	70(2)	20(1)	10(1)	2(1)	2(1)
C(25)	34(2)	42(2)	39(2)	-4(1)	19(1)	-14(1)
C(26)	21(1)	30(1)	19(1)	4(1)	5(1)	-2(1)
C(27)	29(2)	53(2)	55(2)	32(2)	14(1)	9(1)
C(28)	25(1)	23(1)	22(1)	-4(1)	9(1)	-5(1)
C(29)	29(1)	26(1)	20(1)	-6(1)	8(1)	-6(1)
C(30)	29(2)	44(2)	26(1)	-6(1)	7(1)	-15(1)
C(31)	37(2)	26(2)	44(2)	-5(1)	19(1)	-7(1)
C(32)	21(1)	24(1)	34(2)	-4(1)	9(1)	-7(1)
C(33)	28(2)	31(2)	59(2)	5(1)	19(1)	2(1)
C(34)	48(2)	20(2)	74(2)	-8(1)	36(2)	-1(1)
C(35)	62(2)	50(2)	58(2)	-33(2)	44(2)	-26(2)
C(36)	45(2)	54(2)	20(1)	-7(1)	14(1)	-14(2)
C(37)	32(2)	33(2)	35(2)	-1(1)	21(1)	-5(1)

Table 15. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for [$PhBP^{iPr}_3$]FeNAd.

	x	y	z	U(eq)
H(2)	-3380	1141	2994	44
H(3)	-4827	1555	3489	48
H(4)	-4531	2066	4666	33
H(5)	-2740	2194	5316	32
H(6)	-1294	1783	4811	29
H(7A)	-34	501	4307	28
H(7B)	68	1478	4390	28
H(8A)	-2122	375	2576	22
H(8B)	-1803	-127	3316	22
H(9A)	-741	2377	3157	24
H(9B)	-1820	2054	2625	24
H(10A)	2842	2830	3405	62
H(10B)	2506	2028	2917	62
H(10C)	3378	1957	3664	62
H(11)	1175	2430	3578	25
H(12A)	2789	2093	4827	69
H(12B)	1530	2226	4846	69
H(12C)	2216	2960	4592	69
H(13A)	3753	736	4236	57
H(13B)	3231	200	3540	57
H(13C)	3714	-246	4293	57
H(14)	1887	-265	4176	26
H(15A)	2666	-64	5339	50
H(15B)	1615	522	5194	50
H(15C)	2803	906	5232	50
H(16A)	1402	-1846	3606	45
H(16B)	1604	-1095	3101	45
H(16C)	856	-1855	2762	45
H(17)	313	-767	3816	27
H(18A)	-995	-2047	3048	44
H(18B)	-1436	-1403	3561	44
H(18C)	-483	-2033	3897	44
H(19A)	-684	-1413	950	72
H(19B)	363	-1044	1490	72
H(19C)	-486	-438	995	72
H(20)	-784	-1440	2066	60
H(21A)	-2607	-624	1453	67
H(21B)	-2413	-1197	2158	67
H(21C)	-2331	-1581	1391	67
H(22A)	-1288	945	345	42
H(22B)	-93	992	848	42
H(22C)	-678	1813	493	42
H(23)	-1324	804	1466	80
H(24A)	-2568	2151	1183	56
H(24B)	-2659	1401	1718	56
H(24C)	-2838	1244	865	56
H(25A)	665	2627	1004	55
H(25B)	1589	2257	1638	55
H(25C)	1454	3233	1542	55
H(26)	694	2901	2486	28

H(27A)	-265	3907	1773	67
H(27B)	-1109	3371	2111	67
H(27C)	-1022	3239	1285	67
H(29A)	3384	1634	1946	29
H(29B)	3962	957	2516	29
H(30A)	1808	238	663	39
H(30B)	2072	1196	815	39
H(31A)	3556	-544	2288	41
H(31B)	2724	-831	1567	41
H(32)	5215	1421	1808	31
H(33A)	5393	32	2209	45
H(33B)	5733	106	1438	45
H(34)	4554	-1047	1430	53
H(35A)	3254	-705	371	62
H(35B)	4426	-345	319	62
H(36)	3068	688	-49	46
H(37A)	4826	1172	542	37
H(37B)	3908	1762	747	37

Figure 4. Fully labeled drawing of of [PhBP$^{iPr}_3$]Co≡N-*p*-tolyl (**10**)

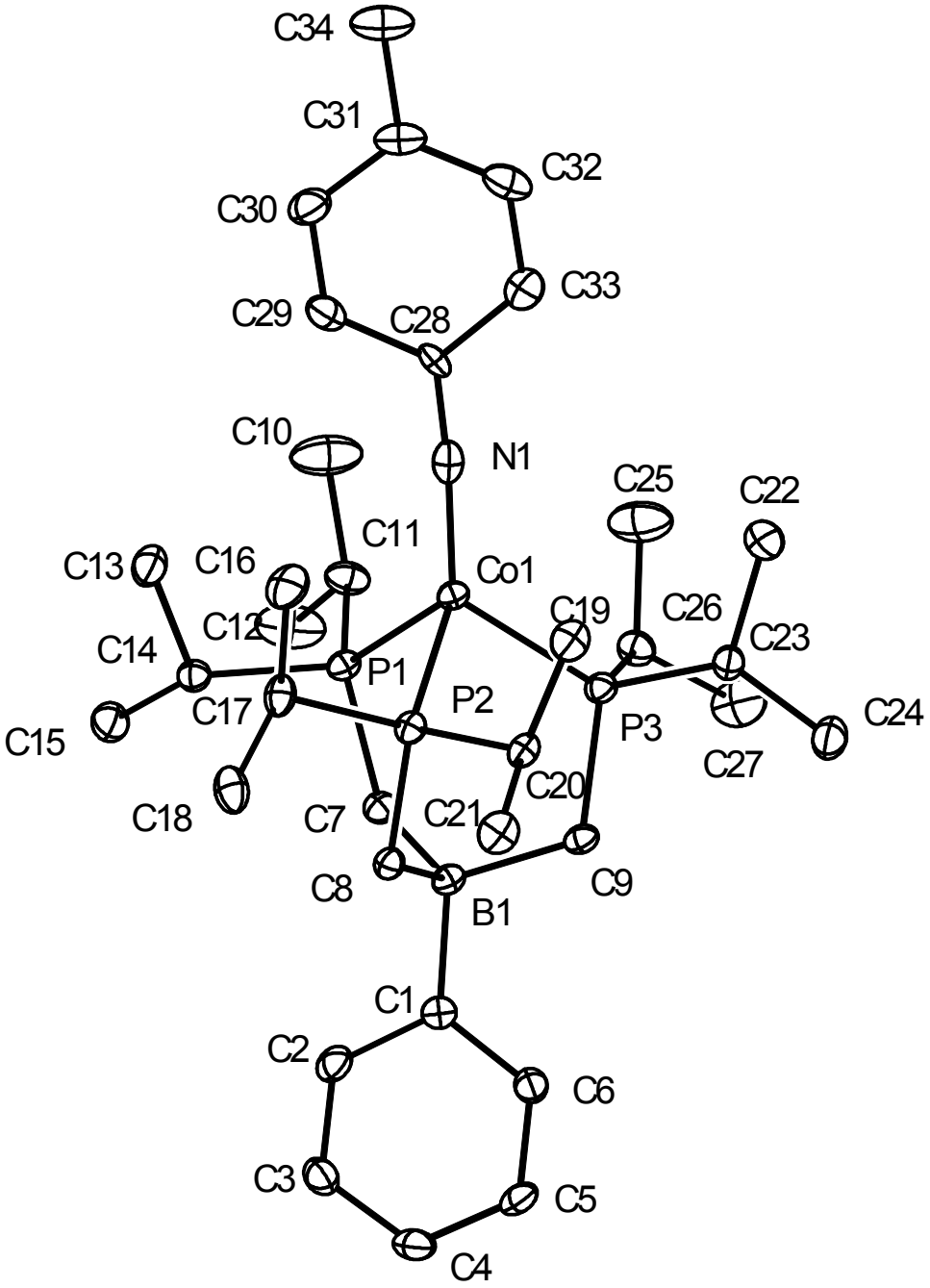

Table 16. Crystal data and structure refinement for [$PhBP^{iPr}_3$]Co≡N-*p*-tol.

Identification code	tab36	
Empirical formula	$C_{41}H_{68}BCoNP_3$	
Formula weight	737.61	
Temperature	96(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 9.8016(9) Å	α= 90°.
	b = 14.8639(13) Å	β= 99.692(2)°.
	c = 28.355(3) Å	γ = 90°.
Volume	4072.1(6) $Å^3$	
Z	4	
Density (calculated)	1.203 Mg/m^3	
Absorption coefficient	0.568 mm^{-1}	
F(000)	1592	
Crystal size	0.22 x 0.22 x 0.36 mm^3	
Theta range for data collection	1.46 to 28.54°.	
Index ranges	-13<=h<=13, -19<=k<=19, -37<=l<=36	
Reflections collected	59668	
Independent reflections	9708 [R(int) = 0.0640]	
Completeness to theta = 28.54°	94.0 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	9708 / 0 / 438	
Goodness-of-fit on F^2	1.620	
Final R indices [I>2sigma(I)]	R1 = 0.0446, wR2 = 0.0838	
R indices (all data)	R1 = 0.0680, wR2 = 0.0871	
Largest diff. peak and hole	0.712 and -0.410 $e.Å^{-3}$	

Special Refinement Details

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Table 17. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [$PhBP^{iPr}_3$]Co≡N-*p*-tol. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Co(1)	9157(1)	3269(1)	1439(1)	12(1)
P(1)	10757(1)	3799(1)	2009(1)	13(1)
P(2)	8286(1)	4605(1)	1256(1)	13(1)
P(3)	10555(1)	3352(1)	904(1)	13(1)
N(1)	8089(2)	2430(1)	1518(1)	16(1)
B(1)	11198(2)	5146(2)	1297(1)	13(1)
C(1)	12115(2)	6052(1)	1252(1)	13(1)
C(2)	12321(2)	6688(1)	1622(1)	19(1)
C(3)	13070(2)	7473(1)	1602(1)	21(1)
C(4)	13653(2)	7660(1)	1201(1)	21(1)
C(5)	13470(2)	7058(1)	827(1)	19(1)
C(6)	12725(2)	6269(1)	853(1)	17(1)
C(7)	11886(2)	4619(1)	1798(1)	14(1)
C(8)	9585(2)	5490(1)	1313(1)	14(1)
C(9)	11222(2)	4473(1)	831(1)	14(1)
C(10)	11076(3)	2110(2)	2481(1)	40(1)
C(11)	11862(2)	2878(1)	2312(1)	20(1)
C(12)	13140(2)	3160(2)	2658(1)	39(1)
C(13)	8929(2)	3862(2)	2677(1)	22(1)
C(14)	10033(2)	4411(1)	2484(1)	15(1)
C(15)	11070(2)	4797(2)	2901(1)	23(1)
C(16)	5757(2)	4295(2)	1597(1)	25(1)
C(17)	6993(2)	4941(1)	1635(1)	17(1)
C(18)	6498(2)	5915(1)	1596(1)	22(1)
C(19)	6474(2)	3847(1)	479(1)	20(1)
C(20)	7399(2)	4665(1)	623(1)	15(1)
C(21)	6619(2)	5536(1)	467(1)	21(1)
C(22)	9177(3)	2016(2)	283(1)	30(1)
C(23)	9688(2)	2987(1)	299(1)	16(1)
C(24)	10496(2)	3168(2)	-109(1)	23(1)
C(25)	11788(3)	1684(2)	1217(1)	36(1)
C(26)	12119(2)	2638(1)	1070(1)	18(1)
C(27)	13153(2)	2605(2)	724(1)	32(1)
C(28)	7152(2)	1843(1)	1599(1)	13(1)
C(29)	6564(2)	1861(1)	2026(1)	22(1)
C(30)	5589(2)	1245(2)	2120(1)	24(1)
C(31)	5127(2)	565(1)	1797(1)	23(1)
C(32)	5683(2)	520(2)	1376(1)	26(1)
C(33)	6660(2)	1134(1)	1279(1)	22(1)
C(34)	4053(2)	-100(2)	1899(1)	33(1)
C(35)	1739(3)	3352(2)	4070(1)	43(1)
C(36)	3100(3)	3186(2)	4233(1)	45(1)
C(37)	3840(3)	3686(2)	4605(1)	52(1)
C(38)	3180(3)	4373(2)	4811(1)	52(1)
C(39)	1825(3)	4535(2)	4643(1)	44(1)
C(40)	1053(3)	4052(2)	4277(1)	40(1)
C(41)	-425(3)	4224(2)	4096(1)	57(1)

Table 18. Bond lengths [Å] and angles [°] for [$PhBP^{iPr}_3$]Co≡N-*p*-tol.

Co(1)-N(1)	1.6668(18)	C(16)-H(16A)	0.9800
Co(1)-P(2)	2.1900(6)	C(16)-H(16B)	0.9800
Co(1)-P(1)	2.1999(6)	C(16)-H(16C)	0.9800
Co(1)-P(3)	2.2127(6)	C(17)-C(18)	1.525(3)
P(1)-C(7)	1.814(2)	C(17)-H(17)	1.0000
P(1)-C(14)	1.862(2)	C(18)-H(18A)	0.9800
P(1)-C(11)	1.863(2)	C(18)-H(18B)	0.9800
P(2)-C(8)	1.819(2)	C(18)-H(18C)	0.9800
P(2)-C(20)	1.859(2)	C(19)-C(20)	1.531(3)
P(2)-C(17)	1.863(2)	C(19)-H(19A)	0.9800
P(3)-C(9)	1.8148(19)	C(19)-H(19B)	0.9800
P(3)-C(26)	1.859(2)	C(19)-H(19C)	0.9800
P(3)-C(23)	1.863(2)	C(20)-C(21)	1.530(3)
N(1)-C(28)	1.314(3)	C(20)-H(20)	1.0000
B(1)-C(1)	1.635(3)	C(21)-H(21A)	0.9800
B(1)-C(9)	1.659(3)	C(21)-H(21B)	0.9800
B(1)-C(7)	1.663(3)	C(21)-H(21C)	0.9800
B(1)-C(8)	1.670(3)	C(22)-C(23)	1.526(3)
C(1)-C(6)	1.402(3)	C(22)-H(22A)	0.9800
C(1)-C(2)	1.401(3)	C(22)-H(22B)	0.9800
C(2)-C(3)	1.385(3)	C(22)-H(22C)	0.9800
C(2)-H(2)	0.9500	C(23)-C(24)	1.532(3)
C(3)-C(4)	1.382(3)	C(23)-H(23)	1.0000
C(3)-H(3)	0.9500	C(24)-H(24A)	0.9800
C(4)-C(5)	1.377(3)	C(24)-H(24B)	0.9800
C(4)-H(4)	0.9500	C(24)-H(24C)	0.9800
C(5)-C(6)	1.391(3)	C(25)-C(26)	1.528(3)
C(5)-H(5)	0.9500	C(25)-H(25A)	0.9800
C(6)-H(6)	0.9500	C(25)-H(25B)	0.9800
C(7)-H(7A)	0.9900	C(25)-H(25C)	0.9800
C(7)-H(7B)	0.9900	C(26)-C(27)	1.525(3)
C(8)-H(8A)	0.9900	C(26)-H(26)	1.0000
C(8)-H(8B)	0.9900	C(27)-H(27A)	0.9800
C(9)-H(9A)	0.9900	C(27)-H(27B)	0.9800
C(9)-H(9B)	0.9900	C(27)-H(27C)	0.9800
C(10)-C(11)	1.501(3)	C(28)-C(33)	1.419(3)
C(10)-H(10A)	0.9800	C(28)-C(29)	1.426(3)
C(10)-H(10B)	0.9800	C(29)-C(30)	1.381(3)
C(10)-H(10C)	0.9800	C(29)-H(29)	0.9500
C(11)-C(12)	1.515(3)	C(30)-C(31)	1.388(3)
C(11)-H(11)	1.0000	C(30)-H(30)	0.9500
C(12)-H(12A)	0.9800	C(31)-C(32)	1.395(3)
C(12)-H(12B)	0.9800	C(31)-C(34)	1.507(3)
C(12)-H(12C)	0.9800	C(32)-C(33)	1.383(3)
C(13)-C(14)	1.528(3)	C(32)-H(32)	0.9500
C(13)-H(13A)	0.9800	C(33)-H(33)	0.9500
C(13)-H(13B)	0.9800	C(34)-H(34A)	0.9800
C(13)-H(13C)	0.9800	C(34)-H(34B)	0.9800
C(14)-C(15)	1.536(3)	C(34)-H(34C)	0.9800
C(14)-H(14)	1.0000	C(35)-C(36)	1.359(4)
C(15)-H(15A)	0.9800	C(35)-C(40)	1.419(4)
C(15)-H(15B)	0.9800	C(35)-H(35)	0.9500
C(15)-H(15C)	0.9800	C(36)-C(37)	1.391(4)
C(16)-C(17)	1.535(3)	C(36)-H(36)	0.9500

C(37)-C(38)	1.388(4)	C(5)-C(6)-C(1)	122.53(19)
C(37)-H(37)	0.9500	C(5)-C(6)-H(6)	118.7
C(38)-C(39)	1.355(4)	C(1)-C(6)-H(6)	118.7
C(38)-H(38)	0.9500	B(1)-C(7)-P(1)	114.61(13)
C(39)-C(40)	1.378(4)	B(1)-C(7)-H(7A)	108.6
C(39)-H(39)	0.9500	P(1)-C(7)-H(7A)	108.6
C(40)-C(41)	1.476(4)	B(1)-C(7)-H(7B)	108.6
C(41)-H(41A)	0.9800	P(1)-C(7)-H(7B)	108.6
C(41)-H(41B)	0.9800	H(7A)-C(7)-H(7B)	107.6
C(41)-H(41C)	0.9800	B(1)-C(8)-P(2)	115.38(14)
N(1)-Co(1)-P(2)	118.77(6)	B(1)-C(8)-H(8A)	108.4
N(1)-Co(1)-P(1)	123.72(6)	P(2)-C(8)-H(8A)	108.4
P(2)-Co(1)-P(1)	92.91(2)	B(1)-C(8)-H(8B)	108.4
N(1)-Co(1)-P(3)	127.57(6)	P(2)-C(8)-H(8B)	108.4
P(2)-Co(1)-P(3)	92.77(2)	H(8A)-C(8)-H(8B)	107.5
P(1)-Co(1)-P(3)	92.09(2)	B(1)-C(9)-P(3)	114.19(13)
C(7)-P(1)-C(14)	103.81(9)	B(1)-C(9)-H(9A)	108.7
C(7)-P(1)-C(11)	107.81(9)	P(3)-C(9)-H(9A)	108.7
C(14)-P(1)-C(11)	106.78(10)	B(1)-C(9)-H(9B)	108.7
C(7)-P(1)-Co(1)	113.41(7)	P(3)-C(9)-H(9B)	108.7
C(14)-P(1)-Co(1)	113.26(7)	H(9A)-C(9)-H(9B)	107.6
C(11)-P(1)-Co(1)	111.25(7)	C(11)-C(10)-H(10A)	109.5
C(8)-P(2)-C(20)	104.96(9)	C(11)-C(10)-H(10B)	109.5
C(8)-P(2)-C(17)	106.76(9)	H(10A)-C(10)-H(10B)	109.5
C(20)-P(2)-C(17)	106.93(9)	C(11)-C(10)-H(10C)	109.5
C(8)-P(2)-Co(1)	113.28(7)	H(10A)-C(10)-H(10C)	109.5
C(20)-P(2)-Co(1)	111.90(7)	H(10B)-C(10)-H(10C)	109.5
C(17)-P(2)-Co(1)	112.48(7)	C(10)-C(11)-C(12)	114.57(19)
C(9)-P(3)-C(26)	104.80(9)	C(10)-C(11)-P(1)	114.60(16)
C(9)-P(3)-C(23)	106.25(9)	C(12)-C(11)-P(1)	116.64(15)
C(26)-P(3)-C(23)	107.21(10)	C(10)-C(11)-H(11)	102.8
C(9)-P(3)-Co(1)	113.94(7)	C(12)-C(11)-H(11)	102.8
C(26)-P(3)-Co(1)	111.99(7)	P(1)-C(11)-H(11)	102.8
C(23)-P(3)-Co(1)	112.09(7)	C(11)-C(12)-H(12A)	109.5
C(28)-N(1)-Co(1)	173.16(15)	C(11)-C(12)-H(12B)	109.5
C(1)-B(1)-C(9)	110.68(16)	H(12A)-C(12)-H(12B)	109.5
C(1)-B(1)-C(7)	107.77(16)	C(11)-C(12)-H(12C)	109.5
C(9)-B(1)-C(7)	109.72(16)	H(12A)-C(12)-H(12C)	109.5
C(1)-B(1)-C(8)	106.61(16)	H(12B)-C(12)-H(12C)	109.5
C(9)-B(1)-C(8)	110.30(16)	C(14)-C(13)-H(13A)	109.5
C(7)-B(1)-C(8)	111.70(16)	C(14)-C(13)-H(13B)	109.5
C(6)-C(1)-C(2)	114.84(18)	H(13A)-C(13)-H(13B)	109.5
C(6)-C(1)-B(1)	124.52(18)	C(14)-C(13)-H(13C)	109.5
C(2)-C(1)-B(1)	120.63(18)	H(13A)-C(13)-H(13C)	109.5
C(3)-C(2)-C(1)	123.24(19)	H(13B)-C(13)-H(13C)	109.5
C(3)-C(2)-H(2)	118.4	C(13)-C(14)-C(15)	109.94(17)
C(1)-C(2)-H(2)	118.4	C(13)-C(14)-P(1)	112.17(14)
C(4)-C(3)-C(2)	119.9(2)	C(15)-C(14)-P(1)	117.18(14)
C(4)-C(3)-H(3)	120.0	C(13)-C(14)-H(14)	105.5
C(2)-C(3)-H(3)	120.0	C(15)-C(14)-H(14)	105.5
C(5)-C(4)-C(3)	119.0(2)	P(1)-C(14)-H(14)	105.5
C(5)-C(4)-H(4)	120.5	C(14)-C(15)-H(15A)	109.5
C(3)-C(4)-H(4)	120.5	C(14)-C(15)-H(15B)	109.5
C(4)-C(5)-C(6)	120.45(19)	H(15A)-C(15)-H(15B)	109.5
C(4)-C(5)-H(5)	119.8	C(14)-C(15)-H(15C)	109.5
C(6)-C(5)-H(5)	119.8	H(15A)-C(15)-H(15C)	109.5

H(15B)-C(15)-H(15C)	109.5	C(26)-C(25)-H(25B)	109.5
C(17)-C(16)-H(16A)	109.5	H(25A)-C(25)-H(25B)	109.5
C(17)-C(16)-H(16B)	109.5	C(26)-C(25)-H(25C)	109.5
H(16A)-C(16)-H(16B)	109.5	H(25A)-C(25)-H(25C)	109.5
C(17)-C(16)-H(16C)	109.5	H(25B)-C(25)-H(25C)	109.5
H(16A)-C(16)-H(16C)	109.5	C(27)-C(26)-C(25)	109.89(18)
H(16B)-C(16)-H(16C)	109.5	C(27)-C(26)-P(3)	117.70(15)
C(18)-C(17)-C(16)	110.56(17)	C(25)-C(26)-P(3)	113.24(15)
C(18)-C(17)-P(2)	116.82(15)	C(27)-C(26)-H(26)	104.9
C(16)-C(17)-P(2)	113.40(15)	C(25)-C(26)-H(26)	104.9
C(18)-C(17)-H(17)	104.9	P(3)-C(26)-H(26)	104.9
C(16)-C(17)-H(17)	104.9	C(26)-C(27)-H(27A)	109.5
P(2)-C(17)-H(17)	104.9	C(26)-C(27)-H(27B)	109.5
C(17)-C(18)-H(18A)	109.5	H(27A)-C(27)-H(27B)	109.5
C(17)-C(18)-H(18B)	109.5	C(26)-C(27)-H(27C)	109.5
H(18A)-C(18)-H(18B)	109.5	H(27A)-C(27)-H(27C)	109.5
C(17)-C(18)-H(18C)	109.5	H(27B)-C(27)-H(27C)	109.5
H(18A)-C(18)-H(18C)	109.5	N(1)-C(28)-C(33)	123.71(19)
H(18B)-C(18)-H(18C)	109.5	N(1)-C(28)-C(29)	121.92(18)
C(20)-C(19)-H(19A)	109.5	C(33)-C(28)-C(29)	114.36(19)
C(20)-C(19)-H(19B)	109.5	C(30)-C(29)-C(28)	122.9(2)
H(19A)-C(19)-H(19B)	109.5	C(30)-C(29)-H(29)	118.5
C(20)-C(19)-H(19C)	109.5	C(28)-C(29)-H(29)	118.5
H(19A)-C(19)-H(19C)	109.5	C(29)-C(30)-C(31)	121.0(2)
H(19B)-C(19)-H(19C)	109.5	C(29)-C(30)-H(30)	119.5
C(19)-C(20)-C(21)	110.45(16)	C(31)-C(30)-H(30)	119.5
C(19)-C(20)-P(2)	112.26(14)	C(30)-C(31)-C(32)	118.0(2)
C(21)-C(20)-P(2)	116.74(14)	C(30)-C(31)-C(34)	120.8(2)
C(19)-C(20)-H(20)	105.5	C(32)-C(31)-C(34)	121.2(2)
C(21)-C(20)-H(20)	105.5	C(33)-C(32)-C(31)	121.4(2)
P(2)-C(20)-H(20)	105.5	C(33)-C(32)-H(32)	119.3
C(20)-C(21)-H(21A)	109.5	C(31)-C(32)-H(32)	119.3
C(20)-C(21)-H(21B)	109.5	C(32)-C(33)-C(28)	122.4(2)
H(21A)-C(21)-H(21B)	109.5	C(32)-C(33)-H(33)	118.8
C(20)-C(21)-H(21C)	109.5	C(28)-C(33)-H(33)	118.8
H(21A)-C(21)-H(21C)	109.5	C(31)-C(34)-H(34A)	109.5
H(21B)-C(21)-H(21C)	109.5	C(31)-C(34)-H(34B)	109.5
C(23)-C(22)-H(22A)	109.5	H(34A)-C(34)-H(34B)	109.5
C(23)-C(22)-H(22B)	109.5	C(31)-C(34)-H(34C)	109.5
H(22A)-C(22)-H(22B)	109.5	H(34A)-C(34)-H(34C)	109.5
C(23)-C(22)-H(22C)	109.5	H(34B)-C(34)-H(34C)	109.5
H(22A)-C(22)-H(22C)	109.5	C(36)-C(35)-C(40)	119.8(3)
H(22B)-C(22)-H(22C)	109.5	C(36)-C(35)-H(35)	120.1
C(22)-C(23)-C(24)	110.64(17)	C(40)-C(35)-H(35)	120.1
C(22)-C(23)-P(3)	113.50(15)	C(35)-C(36)-C(37)	121.3(3)
C(24)-C(23)-P(3)	115.51(14)	C(35)-C(36)-H(36)	119.4
C(22)-C(23)-H(23)	105.4	C(37)-C(36)-H(36)	119.4
C(24)-C(23)-H(23)	105.4	C(38)-C(37)-C(36)	119.4(3)
P(3)-C(23)-H(23)	105.4	C(38)-C(37)-H(37)	120.3
C(23)-C(24)-H(24A)	109.5	C(36)-C(37)-H(37)	120.3
C(23)-C(24)-H(24B)	109.5	C(39)-C(38)-C(37)	118.9(3)
H(24A)-C(24)-H(24B)	109.5	C(39)-C(38)-H(38)	120.6
C(23)-C(24)-H(24C)	109.5	C(37)-C(38)-H(38)	120.6
H(24A)-C(24)-H(24C)	109.5	C(38)-C(39)-C(40)	123.5(3)
H(24B)-C(24)-H(24C)	109.5	C(38)-C(39)-H(39)	118.2
C(26)-C(25)-H(25A)	109.5	C(40)-C(39)-H(39)	118.2

C(39)-C(40)-C(35)	117.1(3)	H(41A)-C(41)-H(41B)	109.5
C(39)-C(40)-C(41)	123.6(3)	C(40)-C(41)-H(41C)	109.5
C(35)-C(40)-C(41)	119.2(3)	H(41A)-C(41)-H(41C)	109.5
C(40)-C(41)-H(41A)	109.5	H(41B)-C(41)-H(41C)	109.5
C(40)-C(41)-H(41B)	109.5		

Table 19. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for [$PhBP^{iPr}_3$]Co≡N-*p*-tol. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^2U^{11} + ...\ + 2\ h\ k\ a\ b\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Co(1)	11(1)	13(1)	13(1)	1(1)	3(1)	-1(1)
P(1)	12(1)	14(1)	14(1)	2(1)	2(1)	-1(1)
P(2)	11(1)	15(1)	13(1)	0(1)	2(1)	0(1)
P(3)	13(1)	13(1)	15(1)	1(1)	4(1)	0(1)
N(1)	15(1)	20(1)	14(1)	0(1)	1(1)	5(1)
B(1)	12(1)	14(1)	13(1)	2(1)	2(1)	-1(1)
C(1)	9(1)	14(1)	17(1)	3(1)	0(1)	2(1)
C(2)	19(1)	20(1)	20(1)	2(1)	9(1)	1(1)
C(3)	23(1)	17(1)	25(1)	-6(1)	9(1)	-3(1)
C(4)	21(1)	14(1)	30(1)	1(1)	8(1)	-3(1)
C(5)	19(1)	21(1)	19(1)	5(1)	9(1)	-2(1)
C(6)	17(1)	17(1)	17(1)	-1(1)	3(1)	0(1)
C(7)	11(1)	15(1)	17(1)	0(1)	3(1)	-2(1)
C(8)	14(1)	15(1)	12(1)	1(1)	3(1)	-1(1)
C(9)	13(1)	13(1)	16(1)	3(1)	5(1)	0(1)
C(10)	31(1)	30(1)	53(2)	21(1)	-6(1)	-5(1)
C(11)	20(1)	16(1)	24(1)	4(1)	-2(1)	0(1)
C(12)	27(1)	29(1)	53(2)	14(1)	-12(1)	-3(1)
C(13)	21(1)	28(1)	17(1)	-2(1)	6(1)	-5(1)
C(14)	14(1)	17(1)	15(1)	-1(1)	3(1)	-1(1)
C(15)	22(1)	27(1)	20(1)	-4(1)	4(1)	-6(1)
C(16)	18(1)	31(1)	29(1)	1(1)	11(1)	-2(1)
C(17)	13(1)	24(1)	15(1)	-2(1)	5(1)	2(1)
C(18)	17(1)	27(1)	23(1)	-7(1)	6(1)	3(1)
C(19)	15(1)	26(1)	18(1)	1(1)	0(1)	-1(1)
C(20)	13(1)	19(1)	12(1)	2(1)	2(1)	0(1)
C(21)	17(1)	25(1)	20(1)	3(1)	0(1)	3(1)
C(22)	44(2)	25(1)	22(1)	-5(1)	7(1)	-12(1)
C(23)	17(1)	17(1)	14(1)	-2(1)	4(1)	-1(1)
C(24)	25(1)	30(1)	17(1)	-3(1)	6(1)	-3(1)
C(25)	28(1)	22(1)	60(2)	12(1)	13(1)	6(1)
C(26)	17(1)	16(1)	21(1)	1(1)	4(1)	3(1)
C(27)	21(1)	31(1)	45(2)	7(1)	12(1)	10(1)
C(28)	12(1)	8(1)	14(1)	-2(1)	-6(1)	7(1)
C(29)	26(1)	17(1)	25(1)	-4(1)	6(1)	-5(1)
C(30)	25(1)	21(1)	28(1)	4(1)	12(1)	-1(1)
C(31)	15(1)	18(1)	35(1)	9(1)	2(1)	-1(1)
C(32)	26(1)	18(1)	31(1)	-1(1)	-3(1)	-5(1)
C(33)	24(1)	23(1)	20(1)	1(1)	4(1)	-1(1)
C(34)	25(1)	25(1)	49(2)	12(1)	2(1)	-8(1)
C(35)	62(2)	33(2)	42(2)	12(1)	29(2)	4(1)
C(36)	54(2)	31(2)	61(2)	11(1)	37(2)	7(1)
C(37)	61(2)	37(2)	65(2)	7(2)	33(2)	1(2)
C(38)	56(2)	46(2)	59(2)	8(2)	23(2)	-5(2)
C(39)	66(2)	29(2)	42(2)	4(1)	27(2)	0(1)
C(40)	46(2)	37(2)	40(2)	22(1)	20(1)	9(1)
C(41)	65(2)	62(2)	44(2)	11(2)	13(2)	6(2)

Table 20. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for [$PhBP^{iPr}_3$]Co≡N-*p*-tol.

	x	y	z	U(eq)
H(2)	11928	6575	1900	22
H(3)	13183	7882	1863	25
H(4)	14171	8196	1185	25
H(5)	13855	7183	548	23
H(6)	12625	5862	592	20
H(7A)	12153	5073	2052	17
H(7B)	12742	4310	1744	17
H(8A)	9315	5930	1052	16
H(8B)	9579	5807	1620	16
H(9A)	12187	4422	773	17
H(9B)	10668	4753	544	17
H(10A)	11651	1567	2510	59
H(10B)	10227	2003	2251	59
H(10C)	10835	2259	2794	59
H(11)	12260	2603	2043	24
H(12A)	12881	3314	2967	58
H(12B)	13559	3685	2530	58
H(12C)	13807	2663	2700	58
H(13A)	9373	3379	2883	32
H(13B)	8290	3600	2409	32
H(13C)	8414	4255	2862	32
H(14)	9536	4943	2321	18
H(15A)	10605	5245	3073	34
H(15B)	11837	5083	2777	34
H(15C)	11429	4311	3121	34
H(16A)	5300	4372	1876	38
H(16B)	6084	3674	1585	38
H(16C)	5098	4428	1305	38
H(17)	7488	4877	1972	21
H(18A)	5806	5989	1306	34
H(18B)	7286	6313	1580	34
H(18C)	6084	6070	1876	34
H(19A)	5605	3916	603	30
H(19B)	6949	3301	612	30
H(19C)	6275	3801	129	30
H(20)	8153	4627	426	18
H(21A)	6460	5578	117	32
H(21B)	7168	6053	603	32
H(21C)	5727	5534	580	32
H(22A)	8482	1925	-5	45
H(22B)	8764	1892	568	45
H(22C)	9958	1607	276	45
H(23)	8833	3364	225	19
H(24A)	11286	2757	-82	35
H(24B)	10829	3791	-90	35
H(24C)	9889	3072	-417	35
H(25A)	11510	1316	929	54
H(25B)	11031	1704	1402	54
H(25C)	12611	1420	1412	54
H(26)	12639	2918	1367	21

H(27A)	14041	2374	891	48
H(27B)	13283	3212	604	48
H(27C)	12800	2208	455	48
H(29)	6855	2317	2255	26
H(30)	5229	1288	2410	29
H(32)	5383	58	1150	31
H(33)	7015	1079	989	27
H(34A)	4496	-681	1987	50
H(34B)	3348	-174	1612	50
H(34C)	3617	123	2163	50
H(35)	1247	2999	3817	52
H(36)	3554	2719	4091	54
H(37)	4789	3558	4718	62
H(38)	3670	4723	5064	63
H(39)	1382	5006	4786	52
H(41A)	-849	4508	4348	85
H(41B)	-894	3654	4002	85
H(41C)	-513	4625	3818	85

Figure 5. Fully labeled drawing of of $\{[PhBP^{iPr}_3]Co(N_2)\}_2Mg(THF)_4$ (**4a**)

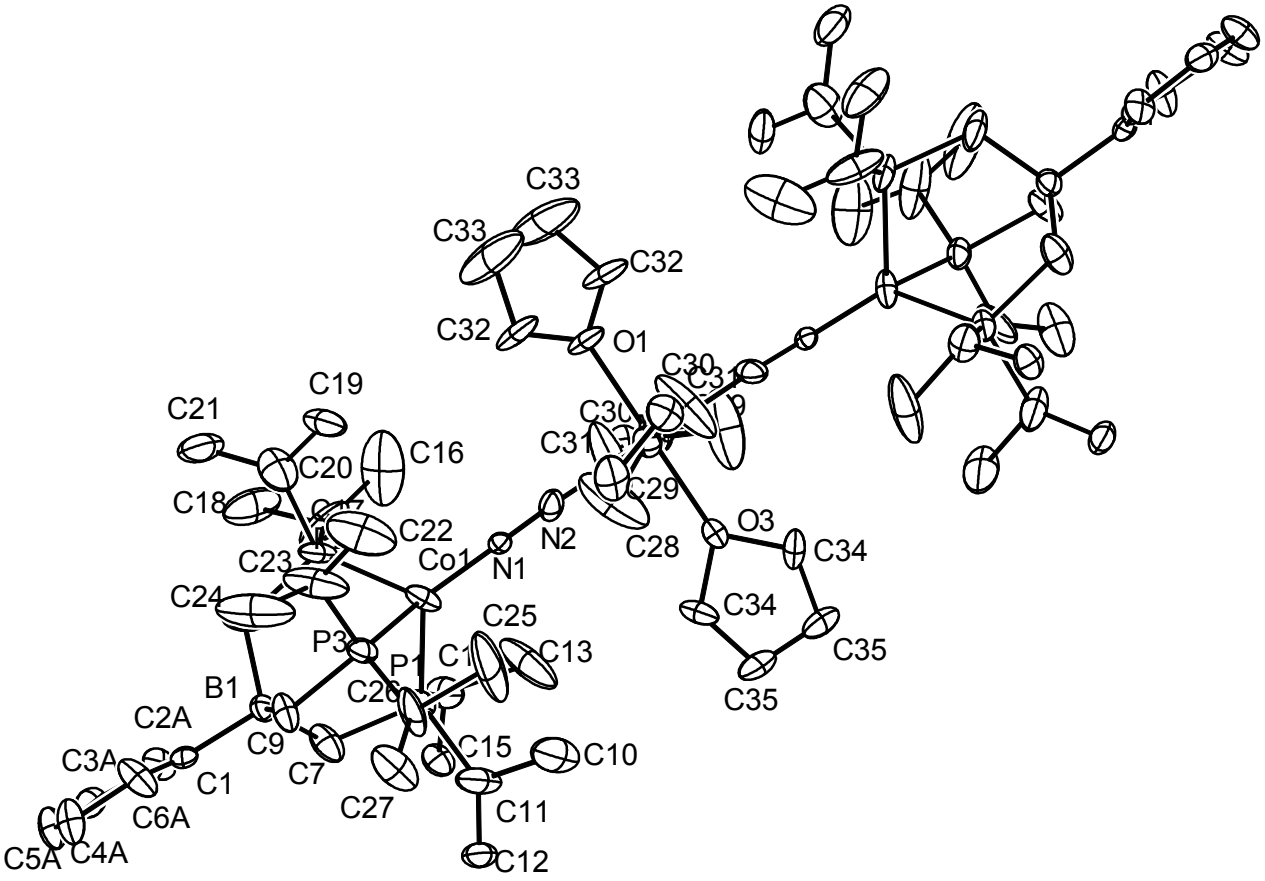

Table 21. Crystal data and structure refinement for $\{[PhBP^{iPr}_3\}Co(N_2)\}_2Mg(THF)_4$.

Identification code	tab32	
Empirical formula	$C_{70}H_{138}B_2Co_2MgN_4O_4P_6$	
Formula weight	1499.51	
Temperature	96(2) K	
Wavelength	0.71073 Å	
Crystal system	Tetragonal	
Space group	I4(1)cd	
Unit cell dimensions	a = 22.4373(8) Å	α= 90°.
	b = 22.4373(8) Å	β= 90°.
	c = 31.7568(17) Å	γ = 90°.
Volume	15987.4(12) Å^3	
Z	8	
Density (calculated)	1.089 Mg/m^3	
Absorption coefficient	0.584 mm^{-1}	
F(000)	5168	
Crystal size	0.16 x 0.25 x 0.33 mm^3	
Theta range for data collection	1.81 to 28.53°.	
Index ranges	-29<=h<=29, -30<=k<=20, -41<=l<=42	
Reflections collected	46683	
Independent reflections	46683 [R(int) = 0.0000]	
Completeness to theta = 28.53°	95.7 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	46683 / 1 / 403	
Goodness-of-fit on F^2	1.591	
Final R indices [I>2sigma(I)]	R1 = 0.1044, wR2 = 0.1538	
R indices (all data)	R1 = 0.2298, wR2 = 0.1681	
Absolute structure parameter	0.028(16)	
Largest diff. peak and hole	0.624 and -0.525 e.Å^{-3}	

Special Refinement Details

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

The structure is disordered in the isopropyl units and the phenyl ring connected to the borate unit in the ligand backbone. The phenyl ring carbon positions are split in two positions and were modeled using half-populations for each atom. The hydrogens were left out of the refinement, as the structure would not converge using standard refinement techniques. Although the thermal ellipsoids are shown (to 25% probability level), the structure is shown to corroborate the proposed connectivity, which is consistent with the complex's combustion analysis. Work is in progress to obtain a higher quality crystal structure of **4a**.

Table 22. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $\{[PhBP^{iPr}_3]Co(N_2)\}_2Mg(THF)_4$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Co(1)	9304(1)	2083(1)	9744(1)	93(1)
N(1)	9553(1)	1374(1)	9720(1)	43(1)
N(2)	9769(2)	908(2)	9701(2)	93(1)
P(1)	9731(1)	2697(1)	10227(1)	77(1)
P(2)	9542(1)	2668(1)	9210(1)	107(1)
P(3)	8376(1)	2465(1)	9802(1)	86(1)
Mg(1)	10000	0	9728(1)	83(1)
B(1)	9041(2)	3588(2)	9747(3)	88(2)
C(1)	8839(2)	4378(2)	9737(2)	95(2)
C(2)	9535(3)	4713(4)	9762(4)	219(3)
C(3)	9330(4)	5448(4)	9702(4)	245(3)
C(4)	8634(3)	5552(3)	9726(3)	135(3)
C(5)	8542(4)	5245(5)	10122(3)	66(3)
C(6)	8593(5)	4674(5)	10094(4)	93(4)
C(5B)	8538(5)	5220(5)	9513(5)	124(7)
C(6B)	8614(4)	4653(4)	9454(4)	103(5)
C(7)	9561(2)	3432(2)	10110(2)	109(2)
C(8)	9300(3)	3452(2)	9250(2)	158(3)
C(9)	8391(2)	3293(2)	9848(2)	105(2)
C(10)	9242(5)	1979(4)	10867(2)	238(5)
C(11)	9434(3)	2596(3)	10759(2)	142(3)
C(12)	9618(3)	3016(3)	11127(2)	123(2)
C(13)	10732(4)	2131(3)	10369(5)	274(6)
C(14)	10498(2)	2719(3)	10239(2)	110(2)
C(15)	10840(2)	3183(2)	10518(2)	105(2)
C(16)	10601(4)	2063(5)	9034(4)	305(9)
C(17)	10446(3)	2704(5)	9099(3)	180(4)
C(18)	10633(4)	3250(4)	8805(2)	203(4)
C(19)	9067(3)	1845(3)	8656(2)	143(3)
C(20)	9264(3)	2493(4)	8661(3)	161(3)
C(21)	9165(5)	2876(3)	8343(2)	221(6)
C(22)	7735(4)	1678(4)	9273(4)	286(6)
C(23)	7862(3)	2347(5)	9367(2)	210(4)
C(24)	7284(3)	2633(5)	9356(3)	361(9)
C(25)	7915(4)	1594(4)	10296(4)	318(8)
C(26)	7949(5)	2300(3)	10276(2)	213(5)
C(27)	7468(3)	2530(3)	10446(2)	149(3)
O(1)	9085(1)	-218(2)	9698(1)	99(1)
C(28)	8830(3)	-743(3)	9617(4)	254(6)
C(29)	8199(2)	-743(2)	9628(2)	123(2)
C(30)	8061(2)	-70(3)	9789(3)	153(2)
C(31)	8634(3)	175(3)	9615(4)	236(6)
O(2)	10000	0	9051(2)	113(2)
C(32)	9987(5)	594(5)	8782(3)	263(7)
C(33)	9948(12)	311(14)	8370(2)	277(14)
O(3)	10000	0	10346(2)	93(2)
C(34)	9834(5)	387(4)	10611(3)	267(7)
C(35)	10007(7)	296(5)	11054(3)	270(9)

Table 23. Bond lengths [Å] and angles [°] for $\{[PhBP^{iPr}_3]Co(N_2)\}_2Mg(THF)_4$.

Co(1)-N(1)	1.687(3)	O(3)-C(34)	1.266(7)
Co(1)-P(2)	2.211(2)	O(3)-C(34)#1	1.266(7)
Co(1)-P(3)	2.2589(14)	C(34)-C(35)	1.472(11)
Co(1)-P(1)	2.2742(18)	C(34)-C(34)#1	1.890(15)
N(1)-N(2)	1.155(3)	C(35)-C(35)#1	1.33(2)
N(2)-Mg(1)	2.103(4)		
P(1)-C(7)	1.734(6)	N(1)-Co(1)-P(2)	116.51(13)
P(1)-C(14)	1.723(5)	N(1)-Co(1)-P(3)	131.85(10)
P(1)-C(11)	1.829(6)	P(2)-Co(1)-P(3)	93.42(7)
P(2)-C(8)	1.845(5)	N(1)-Co(1)-P(1)	117.48(13)
P(2)-C(20)	1.891(7)	P(2)-Co(1)-P(1)	93.16(5)
P(2)-C(17)	2.060(6)	P(3)-Co(1)-P(1)	95.87(5)
P(3)-C(23)	1.821(6)	N(2)-N(1)-Co(1)	174.4(3)
P(3)-C(26)	1.821(8)	N(1)-N(2)-Mg(1)	168.1(4)
P(3)-C(9)	1.862(4)	C(7)-P(1)-C(14)	101.4(3)
Mg(1)-O(3)	1.964(7)	C(7)-P(1)-C(11)	103.5(3)
Mg(1)-N(2)#1	2.103(4)	C(14)-P(1)-C(11)	110.3(3)
Mg(1)-O(1)	2.112(3)	C(7)-P(1)-Co(1)	109.9(2)
Mg(1)-O(1)#1	2.112(3)	C(14)-P(1)-Co(1)	117.02(19)
Mg(1)-O(2)	2.149(7)	C(11)-P(1)-Co(1)	113.2(2)
B(1)-C(9)	1.633(6)	C(8)-P(2)-C(20)	99.5(4)
B(1)-C(7)	1.678(8)	C(8)-P(2)-C(17)	105.3(4)
B(1)-C(8)	1.709(9)	C(20)-P(2)-C(17)	100.1(3)
B(1)-C(1)	1.828(7)	C(8)-P(2)-Co(1)	116.25(19)
C(1)-C(6B)	1.204(7)	C(20)-P(2)-Co(1)	120.2(3)
C(1)-C(6)	1.424(12)	C(17)-P(2)-Co(1)	113.1(3)
C(1)-C(2)	1.737(8)	C(23)-P(3)-C(26)	105.3(4)
C(2)-C(3)	1.723(9)	C(23)-P(3)-C(9)	102.4(4)
C(3)-C(4)	1.581(10)	C(26)-P(3)-C(9)	98.6(3)
C(4)-C(5B)	1.028(10)	C(23)-P(3)-Co(1)	117.7(2)
C(4)-C(5)	1.450(11)	C(26)-P(3)-Co(1)	118.4(3)
C(5)-C(6)	1.289(14)	C(9)-P(3)-Co(1)	111.68(17)
C(5B)-C(6B)	1.297(12)	O(3)-Mg(1)-N(2)#1	92.27(18)
C(10)-C(11)	1.490(9)	O(3)-Mg(1)-N(2)	92.27(18)
C(11)-C(12)	1.559(8)	N(2)#1-Mg(1)-N(2)	175.5(4)
C(13)-C(14)	1.477(9)	O(3)-Mg(1)-O(1)	92.59(15)
C(14)-C(15)	1.567(7)	N(2)#1-Mg(1)-O(1)	90.75(13)
C(16)-C(17)	1.494(13)	N(2)-Mg(1)-O(1)	89.05(13)
C(17)-C(18)	1.598(10)	O(3)-Mg(1)-O(1)#1	92.59(15)
C(19)-C(20)	1.520(9)	N(2)#1-Mg(1)-O(1)#1	89.05(13)
C(20)-C(21)	1.347(8)	N(2)-Mg(1)-O(1)#1	90.75(13)
C(22)-C(23)	1.556(11)	O(1)-Mg(1)-O(1)#1	174.8(3)
C(23)-C(24)	1.447(8)	O(3)-Mg(1)-O(2)	180.000(1)
C(25)-C(26)	1.587(10)	N(2)#1-Mg(1)-O(2)	87.73(18)
C(26)-C(27)	1.312(8)	N(2)-Mg(1)-O(2)	87.73(18)
O(1)-C(28)	1.334(7)	O(1)-Mg(1)-O(2)	87.41(15)
O(1)-C(31)	1.369(5)	O(1)#1-Mg(1)-O(2)	87.41(15)
C(28)-C(29)	1.417(7)	C(9)-B(1)-C(7)	113.7(5)
C(29)-C(30)	1.625(9)	C(9)-B(1)-C(8)	114.3(6)
C(30)-C(31)	1.502(8)	C(7)-B(1)-C(8)	111.1(5)
O(2)-C(32)#1	1.583(9)	C(9)-B(1)-C(1)	100.1(3)
O(2)-C(32)	1.583(9)	C(7)-B(1)-C(1)	112.7(4)
C(32)-C(33)	1.457(19)	C(8)-B(1)-C(1)	104.0(4)
C(33)-C(33)#1	1.42(5)	C(6B)-C(1)-C(6)	101.0(9)

C(6B)-C(1)-C(2)	100.9(7)	C(21)-C(20)-P(2)	127.9(6)
C(6)-C(1)-C(2)	96.3(6)	C(19)-C(20)-P(2)	107.7(6)
C(6B)-C(1)-B(1)	127.9(7)	C(24)-C(23)-C(22)	105.0(8)
C(6)-C(1)-B(1)	122.4(7)	C(24)-C(23)-P(3)	121.4(6)
C(2)-C(1)-B(1)	101.3(4)	C(22)-C(23)-P(3)	113.8(7)
C(3)-C(2)-C(1)	99.7(6)	C(27)-C(26)-C(25)	109.6(8)
C(2)-C(3)-C(4)	113.6(7)	C(27)-C(26)-P(3)	133.9(6)
C(5B)-C(4)-C(5)	101.2(12)	C(25)-C(26)-P(3)	105.2(5)
C(5B)-C(4)-C(3)	93.9(10)	C(28)-O(1)-C(31)	102.3(5)
C(5)-C(4)-C(3)	96.4(7)	C(28)-O(1)-Mg(1)	129.1(4)
C(6)-C(5)-C(4)	113.6(9)	C(31)-O(1)-Mg(1)	125.4(4)
C(5)-C(6)-C(1)	123.6(10)	O(1)-C(28)-C(29)	115.1(5)
C(4)-C(5B)-C(6B)	141.3(13)	C(28)-C(29)-C(30)	101.4(5)
C(1)-C(6B)-C(5B)	116.7(11)	C(31)-C(30)-C(29)	93.5(6)
P(1)-C(7)-B(1)	119.9(3)	O(1)-C(31)-C(30)	109.1(6)
B(1)-C(8)-P(2)	109.6(4)	C(32)#1-O(2)-C(32)	114.7(10)
B(1)-C(9)-P(3)	113.9(3)	C(32)#1-O(2)-Mg(1)	122.7(5)
C(10)-C(11)-C(12)	117.7(6)	C(32)-O(2)-Mg(1)	122.7(5)
C(10)-C(11)-P(1)	115.7(5)	C(33)-C(32)-O(2)	96.9(13)
C(12)-C(11)-P(1)	121.4(5)	C(33)#1-C(33)-C(32)	114.8(8)
C(15)-C(14)-C(13)	105.2(5)	C(34)-O(3)-C(34)#1	96.5(9)
C(15)-C(14)-P(1)	121.4(4)	C(34)-O(3)-Mg(1)	131.7(5)
C(13)-C(14)-P(1)	109.6(6)	C(34)#1-O(3)-Mg(1)	131.7(5)
C(16)-C(17)-C(18)	126.6(8)	O(3)-C(34)-C(35)	117.6(8)
C(16)-C(17)-P(2)	102.5(6)	O(3)-C(34)-C(34)#1	41.7(5)
C(18)-C(17)-P(2)	112.9(6)	C(35)-C(34)-C(34)#1	76.6(5)
C(21)-C(20)-C(19)	123.7(6)	C(35)#1-C(35)-C(34)	97.6(7)

Symmetry transformations used to generate equivalent atoms:
#1 x, y, z #2 -x+1/2, -y+1/2, z+1/2 #3 -y, x+1/2, z+1/4 #4 y+1/2, -x, z+3/4
#5 x, -y, z+1/2 #6 -x+1/2, y+1/2, z #7 -y, -x+1/2, z+3/4 #8 y+1/2, x, z+1/4
#9 x+1/2, y+1/2, z+1/2 #10 -x+1, -y+1, z+1 #11 -y+1/2, x+1, z+3/4 #12 y+1, -x+1/2, z+5/4
#13 x+1/2, -y+1/2, z+1 #14 -x+1, y+1, z+1/2 #15 -y+1/2, -x+1, z+5/4 #16 y+1, x+1/2, z+3/4

Table 24. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for $\{[PhBP^{iPr}_3]Co(N_2)\}_2Mg(THF)_4$. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^2U^{11} + ...\ + 2\ h\ k\ a\ b\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Co(1)	90(1)	60(1)	129(1)	-36(1)	28(1)	-8(1)
N(1)	30(2)	76(3)	24(2)	-7(2)	15(2)	16(2)
N(2)	107(4)	96(3)	77(3)	8(4)	23(3)	31(2)
P(1)	72(1)	60(1)	97(1)	-4(1)	32(1)	0(1)
P(2)	119(2)	101(1)	101(1)	-55(1)	40(1)	-28(1)
P(3)	94(1)	65(1)	97(1)	-24(1)	0(1)	-7(1)
Mg(1)	88(2)	92(2)	69(2)	0	0	37(2)
B(1)	101(5)	30(3)	134(6)	-15(4)	60(6)	-16(3)
C(1)	148(5)	100(4)	37(3)	-3(4)	-8(4)	-85(4)
C(4)	184(8)	110(5)	112(5)	13(6)	-24(7)	56(5)
C(5B)	74(7)	47(7)	249(18)	-75(8)	-116(9)	18(6)
C(6B)	110(8)	4(5)	195(12)	36(6)	-158(8)	24(5)
C(7)	95(4)	74(4)	157(6)	-12(4)	-59(4)	-13(3)
C(8)	307(9)	25(3)	141(6)	-4(4)	162(6)	8(4)
C(9)	132(4)	37(3)	147(6)	4(4)	-40(4)	-4(3)
C(10)	484(15)	144(8)	85(6)	36(5)	-1(7)	39(8)
C(11)	258(9)	81(5)	89(5)	9(4)	35(5)	-27(5)
C(12)	150(6)	104(5)	114(5)	-49(4)	-2(4)	-16(4)
C(13)	204(9)	113(6)	506(18)	-116(9)	-177(11)	62(6)
C(14)	74(4)	138(6)	119(5)	-73(4)	-71(4)	13(4)
C(15)	71(4)	73(4)	170(6)	-26(4)	-7(4)	-16(3)
C(16)	136(7)	362(15)	418(18)	281(14)	116(9)	166(9)
C(17)	52(4)	355(13)	133(7)	65(8)	-10(4)	-79(6)
C(18)	266(9)	254(9)	88(6)	7(6)	83(6)	-108(8)
C(19)	171(7)	96(5)	163(7)	-47(5)	-23(5)	22(5)
C(20)	136(5)	161(8)	186(9)	-25(7)	-94(6)	-36(5)
C(21)	538(18)	96(5)	29(4)	23(4)	-13(7)	-96(8)
C(22)	167(8)	308(12)	384(14)	-131(12)	-59(8)	-140(9)
C(23)	86(5)	359(12)	184(8)	-164(8)	-84(5)	37(7)
C(24)	78(5)	576(18)	430(14)	-375(13)	-125(7)	210(8)
C(25)	317(12)	177(8)	458(18)	175(10)	284(13)	67(8)
C(26)	369(12)	109(6)	161(8)	86(5)	131(8)	141(7)
C(27)	118(6)	113(5)	216(8)	-34(5)	93(5)	-6(5)
O(1)	93(3)	116(3)	87(3)	-12(3)	59(3)	27(2)
C(28)	79(5)	79(5)	604(19)	-52(9)	64(8)	1(4)
C(29)	60(4)	92(4)	216(7)	7(5)	36(4)	2(3)
C(30)	54(3)	196(6)	209(7)	19(6)	2(6)	-31(5)
C(31)	58(4)	194(8)	457(18)	96(9)	-22(8)	47(5)
O(2)	104(4)	149(6)	86(5)	0	0	2(3)
C(32)	514(19)	173(12)	101(8)	90(9)	-21(9)	14(11)
C(33)	218(13)	570(50)	42(4)	17(10)	-7(8)	100(20)
O(3)	139(6)	86(5)	55(4)	0	0	54(4)
C(34)	600(20)	117(7)	83(7)	-51(5)	-16(9)	134(9)
C(35)	540(20)	176(11)	99(8)	-32(6)	-66(12)	64(14)